1.0

4.5
5.0
5.6
6.3

2.8

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3.2

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1.8

1.25

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MICROCOPY RESOLUTION TEST CHART
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MATERIALS NOTE 133

SURFACE MODIFICATION BY PHYSICAL VAPOUR DEPOSITION

by

R.B. NETHERCOTT

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SURFACE MODIFICATION BY PHYSICAL VAPOUR DEPOSITION

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SUMMARY

The combination of properties required from modern materials has led to
the design of systems in which the bulk physical properties are obtained from
the main structure and the required surface properties are provided by a
surface coating.

One important class of techniques for coating surfaces is Physical Vapour
Deposition (P.V.D.). This class includes the processes of Evaporation,
Sputtering, Ion-Plating and Ion-Implantation, by which a wide variety of
materials can be applied to different substrates.

A review is given of the above P.V.D. processes and the resultant coating
morphologies. Areas of present and future applications are discussed for which
surface governed properties such as corrosion, wear, bearing and tool life,
fatigue life and surface hardening can be beneficially modified.

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1 INTRODUCTION

The combination of properties required from modern materials are such that often they can only be achieved by developing appropriate "composite" materials. One method of achieving a suitable combination of properties, such as high temperature strength and corrosion resistance, is to select a high strength material and impart the required corrosion properties at the surface by suitable deposition processes. An example of such a composite system is that of chrome steels having a stable adhering protective surface oxide resulting from the implantation of Y ions [1].

Surface modifications can be achieved by a wide variety of coating techniques Table 1. The last four processes in the table, viz. evaporation, sputtering, ion plating and ion implantation, are linked by the generic description of Physical Vapour Deposition (P.V.D.). P.V.D. processes are designed to transfer a wide range of coating materials to a similarly wide range of substrate materials and are characterised by an atom-by-atom transfer mode, although sputtering can involve clusters of atoms. If chosen correctly, these P.V.D. coatings can have a marked effect on the physical and chemical properties of the new composite material, e.g. on the corrosion resistance, wear rate, friction, strength and fatigue properties of a system. Marked changes in these properties are induced by changes to the structure and composition of the surface to a depth as small as 0.2 micrometres. However, the coating processes can sometimes degrade the properties of the base material as, for instance, can happen with hydrogen embrittlement of steels during electroplating.

An indication of the work being done on new methods of surface protection and coating is given in Table 2 [2], (traditional coating processes were not considered for the compilation).

The processes of evaporation, sputtering, ion plating and ion implantation will be described below, followed by a discussion of the morphology of the coatings produced by each process and a review of applications.

2 DEPOSITION PROCESSES

2.1 EVAPORATION

In this process, vapourisation of the material to be deposited is induced by such means as radiation, eddy currents, laser, electron beam bombardment and electrical discharge (Table 3). The thermally evaporated atoms have an energy of between 0.2 and 1 eV, and follow a collisionless, line-of-sight path prior to condensation on the substrate, which is usually at ground potential, Fig. 1. The vacuum is between 10^{-5} to 10^{-6} mm of mercury (10^{-3} to 10^{-4} Pa) [3].

As seen in Fig. 1, the line-of-sight path causes the deposit thickness and vapour beam density to be greatest at the centre line, with the beam density down to 50% at 30 degrees from the substrate-source axis. This non-uniformity can be minimised by specimen scanning techniques or by using a gas pressure of approximately 0.2 mm of mercury (26.6 Pa) which causes vapour collisions to deviate the atoms from the straight line path.

COATING MATERIAL
Any pure metal and many alloys and compounds are suitable for evaporation. The source can be either a rod of the alloy or separate rods of each alloy component; for alloy deposition, the evaporation rate from each source rod needs to be monitored. If a reactive gas is introduced during evaporation, e.g. N_2 at a pressure of 250 Pa, a compound may be plated e.g. TiN or Ti_2N [4, 5].

SUBSTRATE
Substrates of metal, ceramics, glass, plastic or paper must be of simple geometry.

DEPOSITION RATE
Relatively high rates of deposition of 10 to 4500 micrometres/hour can be achieved.

ADHESION
The adhesion of the coating is very dependent on the substrate temperature, with better adhesion being achieved at higher temperatures and after sputter-cleaning of the substrate.

2.2 SPUTTERING

The sputtered coating (Table 3) is formed from vapour atoms dislodged from the source rod by knock-on collisions with gas ions (usually argon) produced in a glow discharge [6], usually in a planar diode configuration, Fig. 2. The source is usually the cathode and the substrate forms the anode [6]. The gas discharge is established at 2 to 5 kV with a current density of approximately 0.5 mA/cm^2 [8], and a gas pressure of between 0.02 to 0.15 mm of mercury (2.6 to 16.5 Pa). Sputtering is a relatively inefficient process for producing vapour atoms due to the poor yield per incident argon ion. The energy of the incident argon ions must exceed the lattice binding energy of approximately 10 eV to dislodge atoms, but the ions usually have an energy distribution in the range 0.1 to 100 eV [8, 9].

Because of the collisions undergone by the source atoms, the uniformity of coating is much better than that obtained by thermal evaporation techniques, and good coverage is obtained except in deep recesses.

There is a greater yield of sputtered atoms if the incident ions impinge at angles of between 50 and 80 degrees from the normal. For these angles, there is then a greater chance of a cascade closer to the surface since energy dissipation from the incident beam occurs closer to the surface at higher angles of incidence [9]. However, in some instances, a maximum yield is obtained at 30 degrees [10]. At higher angles the incident ions bounce off the surface and the yield of sputtered atoms decreases [11].

The parameters important in sputtering include:
Mass of the gas ion compared with the source atoms,
Ion energy, [12]
Ion incident angle,
Temperature of source,

Ion current,
Gas pressure,
Type of gas and its reactivity,
Impurities in source.

Sputtering does not depend on the vapour pressure of the source elements and is considered a "cold process", with the source atoms being dislodged by a "knock-on" rather than an evaporative process. It should be noted, however, that in a sputtering discharge, only a small percentage of the gas atoms are ionized, with the result that the cathode and other surfaces in the system are being continuously bombarded by non-ionised thermal atoms and molecules, thereby causing heating of the substrate.

COATING MATERIAL

Any metal or non-metal including insulators, glasses, inorganic compounds e.g. P.T.F.E. [13], and even cattle bone may be sputtered [6]. Sputtering is especially useful for high melting point, high thermal conductivity metals such as Cu, Pt, Au, and Ag, which are not as easily thermally evaporated. Regardless of chemical complexity, any solid material can be sputtered with the same stoichiometry as the bulk material. Sources can be layered to achieve a variation in sputtered coating composition.

SUBSTRATE

Non-gassing surfaces of metal, glass, ceramic, plastic or paper are suitable, and composite metal - non-metal interfaces can be achieved. The substrate is often water-cooled to avoid thermal heating and bulk surface diffusion, if stoichiometry of sputtered compounds is required. Cooling of the substrate is also required at high deposition rates [6].

DEPOSITION RATE

Deposition rate for sputtering is lower than for evaporation, with a sputtering yield of between 1 to 50 source atoms per incident argon ion [6].

Rates of 0.25 to 250 micrometres/hour are obtained with coatings of thickness up to 6.0 mm.

ADHESION

Coating adhesion to most substrates is good, and can be excellent, if the substrate has been sputter-cleaned [14]. High arrival energies of the sputtered material also aid cleaning and adhesion.

2.3 ION PLATING

Ion plating (Table 3) forms coatings whose properties are similar to those of the bulk coating material. During ion plating, Fig. 3, the source material is vapourised by heating, as for evaporation, but the atoms then pass through an inert gas discharge (such as argon) in which some of the vapourised atoms become ionised and accelerate in an electric field[3, 15] to bombard the substrate (cathode), which is biased to a high d.c. or r.f. negative potential (2 to 5 kV). The gas pressure is 5 to 200 x 10^{-3} mm Hg (0.5 to 30 Pa). The source can be vaporised by many different methods of heating, e.g. resistively heated filaments, electron-beam heated crucibles, r.f. heating, sputtering cathodes, metal bearing gases (chemical ion plating).

As can be seen from Fig. 4, ion plating has much higher throwing power than evaporation and sputtering, and enables more uniform coatings on complex, intricately shaped substrates. It is uncertain whether the higher throwing power is due to the ions following the electric field lines to the back and internal surfaces or whether it is due mainly to gas scattering effects [16].

The gas discharge also causes the substrate to be sputter-cleaned due to bombardment by high energy gas ions [17, 18]. Another surface-cleaning technique used in surface-coating is that of reactive plasma-cleaning in which a reactive species in the discharge gas chemically reacts with the substrate to give a volatile species [8, 18, 19]. Halide gases have been found to be useful in cleaning metal surfaces by this means, e.g. Ti can be sputter-cleaned by Ar and then reactive plasma cleaned using HCl or CCl_4

[5]

[8]. Oxygen plasma may be used to clean insulators and gold surfaces and may also be used to remove carbon and hydrocarbon contamination from metals which form passive oxides. Difficulties may arise if the reactive gases present form compound films of the reaction products (reactive ion plating) [20], e.g. Ti evaporated in a nitrogen gas forms Ti-nitrides [5, 17]. This situation may or may not be desirable. It should be noted that contaminant gases are often more reactive in the discharge than in the simple gaseous state. A major factor in the recent development of ion plating is the recognition of the better adhesion between incompatible substrates compared with the previously mentioned sputtering and evaporation techniques.

COATING MATERIAL

The technique is most effective for pure metals, but virtually any material that can be successfully evaporated without change in chemical composition can be ion plated [6, 15]. Alloys, compounds, and ceramics can be successfully ion plated [17, 15]. The coatings are coherent and can be fully dense [6].

SUBSTRATE

Ion plating is usually reserved for conducting substrates, but it can sometimes be applied to non-conductors. Thus, metals can be plated onto plastics provided care is taken to avoid heating of the substrate [17, 15]. The high energy and the gaseous ion sputter-cleaning cause heating that the substrate must be able to withstand. The substrate can be water-cooled to help alleviate the heating or else it can be surrounded by a heated auxiliary cathode to support the discharge.

The good to excellent adhesion of ion plating allows unusual materials combinations that are not readily achievable by other processes, e.g. metal to plastic and ceramic to metal [17]. Large work pieces are also readily plated.

DEPOSITION RATE

As with sputtering, the deposition rate during ion plating is reduced by sputtering of the substrate. This process not only cleans and removes substrate and impurity atoms, but also the recently plated source atoms. The overall plating rate is slightly higher than sputtering at approximately 5 to 1500 micrometres/hour.

ADHESION

Excellent adhesion is achieved, often between normally incompatible coatings and substrates, because the sputter-cleaning removes oxides and other layers that may impair adhesion and intimate contact of the plated ions with the substrate. It is important that the sputter-cleaning bombardment should continue throughout deposition without interruption, although for the coating to form, the deposition rate needs to exceed the sputtering rate. Control of the plating components may improve adhesion, e.g. an initial thin layer of Al on steel improves the adhesion of a final coating of Al_2O_3 [17]. The interdiffusion of coating and substrate caused by the discharge heating enhances adhesion as does the production of near surface vacancies by the impinging ions. Heating of the substrate may result in a diffusion gradient rather than an abrupt interface.

2.4 ION IMPLANTATION

Ion implantation is a much more expensive process than the techniques discussed previously, and provides lower rates of material deposition [17]. Some of the typical technical parameters and economic factors relating to ion implantation are given in Table 3 and Table 4. Typical systems with an accelerating voltage of 400 kV cost more than 300,000(1980) dollars [21, 17], although it seems that with current technology, the cost of an implant for the typical doses used for improving wear and corrosion resistance is estimated to be approximately 30cents/cm^2, and it could eventually fall to 1 cent/cm^2[17, 22]. Ion implantation has similarities with ion plating, but the ionised source material is introduced into the substrate by accelerating the ions to a much higher energy (at least 10kV and usually 100kV or higher) thus embedding them in the substrate, Fig. 5 [23]. The ionised atoms can be formed in many ways, including electrical discharge in a gas of chosen atoms, plasma discharge, volatilisation of

metal chlorides, dissociation of solids in a vacuum, field ionisation, and laser heating [11].

For beam handling, ions of different mass cannot be separated by an electrostatic device and must be manipulated by a combination of electric and magnetic fields or by an a.c. electric field [11]. The vacuum in the system is approximately 8×10^{-6} to 10^{-11} mm Hg (10^{-3} to 1.5×10^{-9} Pa) [24, 25]. Ion implantation has poor coverage due to line-of-sight passage of the ions.

The impinging ions lose their energy by interaction with the substrate atoms and penetrate to a depth of 0.01 to 1 micrometres with an approximately Gaussian distribution of ion concentration with depth [26, Ref 8]. The result is a thin layer at the surface of the substrate of different alloy composition with altered chemical properties. A schematic representation of the ion paths is shown in Fig. 6. The final shape of the resulting distribution of the ions will depend on processing parameters such as type of ion, type of substrate, accelerating voltage and subsequent heat-treatment. The variation in mean range of ions in aluminium by more than a factor of 100 is shown in Fig. 7 for different types and energy of the impinging ions. The depth of the implantation and the varying alloy composition with depth is a major difference compared with the previous methods of vapour deposition. The first three methods produce surface coatings with observable demarcation between coatings and substrates. Ion implantation, however, produces an intimate joining of the source atoms and substrate. The alloying need not occur as a stable system, but may be in a metastable condition, not formable by conventional alloying or surface treatments.

Because of the depth of penetration, ion implantation processes do not reqire the high temperatures sometimes necessary for the diffusion of coatings into the substrate [17].

Results of ion implantation are often so unusual, with the formation of exotic and unusual alloy compositions, that properties and possible applications have not yet been fully explored [17]. Metallurgical and solid state theory is often unable to provide a guide to expected developments.

A major motivation for exploitation of ion implantation is a reduction in the use of certain strategic or expensive metals such as chromium and cobalt [17]. Often components whose surfaces have been modified by ion implantation will show properties similar to those of bulk-alloyed metals, particularly for corrosion or wear resistance.

COATING MATERIAL
The technique is most widespread in the semiconductor industry for doping processes. Almost all elements, and many alloys and compounds, can be implanted.

SUBSTRATE
Semiconductors, insulators and metals are all suitable substrates. Since ion implantation is a low temperature process and the substrate is not heated, minimal dimensional changes result.

DEPOSITION RATE
Ions penetrate to approximately 1000A, with a Gaussian composition profile [17]. This profile has the effect of forming an alloy of source - substrate material without a definite demarcation of the interface, and composition varying with depth. Lower rates of deposition are obtained compared with ion plating [17]. The number of ions implanted per unit area (fluence) in metallurgical applications is 10^{14} to 10^{18} ions/cm^2 compared with 10^{12} to 10^{16} ions/cm^2 for semiconductor applications. In the semiconductor industry, work pieces are usually wafers of area less than 100 cm^2 [17]. Such metallurgical applications as ball-bearings or fasteners would be of similar area and present equipment is therefore still applicable. However, if large scale applications, such as to improve corrosion-resistance of structures, were contemplated, major equipment redesign would be necessary.

[7]

ADHESION

The absence of discontinuous surface layers gives excellent adhesion. Even very thin interfacial regions (10 to 1000A) may be effective in distributing interfacial stresses [8].

An example of increased adhesion arising from ion implantation occurs when an evaporated aluminium film on glass is bombarded with argon ions at 100kV. The aluminium film shows increased adhesion to the glass, probably due to the formation of a glass-to-metal transition layer at the interface. The force per unit area required to strip the aluminium film increases from 0.35 to approximately 21 MPa, Fig. 8 [27].

3 COATING MORPHOLOGY

For a coating to be formed, an atom or ion arriving at the substrate surface must exchange sufficient energy to become bound to the surface [3]. The microstructure of the deposit is therefore affected by those thermally activated processes that influence surface mobility and by the density of suitable bonding sites on the surface. The surface temperature and atom energy are two of the more important parameters determining the atom-binding conditions and thus the coating microstructure.

3.1 EVAPORATED AND SPUTTERED COATINGS

The coating morphology is related to deposition temperature [28], e.g. at low temperatures $(-196^{\circ}C)$, there is more likelihood of amorphous phases being formed than at higher temperatures [29]. The different morphologies tend to form at substrate temperatures in the ranges $T < 0.3\ T_m < 0.45\ T_m < T_m$, where T_m is the melting point of the source [28]. The transition between ranges is not sharp, but gradual. Qualitatively, the ranges have been seen in metals and compounds e.g. Ni, Ti, W, Al_2O_3, ZrO_2, Fe-Nb-C, with the range limits tending to occur at higher temperatures for complex compounds and alloys [3].

The forms of microstructure are shown schematically in Fig. 9 and the effect of process parameters can be summarised (Table 5) as follows:-

$T < 0.3T_m$

Surface diffusion is low and the absorbed atoms tend to join as small islands that grow out from the substrate as elongated and tapered crystallites [3, 28]. These crystallites eventually coalesce, without a pronounced boundary zone and with much intercrystalline porosity, to form a complete surface-coating [31]. Dislocation density is also high [32].

$0.3T_m < T < 0.45T_m$

In the intermediate region, there is sufficient atom mobility for the low temperature crystallite type of coating to form into elongated columnar grains with definite crystal structure and grain boundaries [3]. The grain diameter increases with temperature. Dislocation density is much lower than in the microstructures formed at lower temperatures and the dislocations are mainly in the grain boundaries [32].

$0.45T_m < T$

The high temperature region has a fine-grained, equiaxed structure similar to a bulk metal after an equilibrium anneal [3].

Figs. 10 and 11 show optical micrographs of evaporated titanium deposits produced at different substrate temperatures. The changes in microstructure and porosity can be understood in terms of the increased atom mobility as the temperature increases. The different morphologies shown schematically in Fig. 9 can be identified in the optical micrographs. For titanium $(T_m=1945K)$, the morphology changes occur at approximately 580 and 950K.

If the energy of the source atoms is increased by ionisation, as occurs during sputtering, refinement of coating morphology occurs at lower substrate temperatures [3]. This refinement could be caused by an enhanced diffusion rate and/or a higher nucleation rate and consequent smaller grain size. Thus, as the bias voltage of the substrate and therefore the ion energy is increased, the degree of refinement of the coating is increased for a given substrate deposition temperature. An increase in gas pressure has the reverse effect because the mean free path, and hence the incident ion energy, is effectively reduced, resulting in a coarser microstructure.

As with the general crystal structure of the coating, other properties are also affected by deposition parameters.

(a) Texture

The texture is generally affected by deposition temperature, and is more highly oriented at lower temperatures [3]. The elongated grains at low

[9]

temperatures (shown in Figs. 9 to 11) are reported to possess some form of preferred crystallographic orientation [28]. In Be [3], the texture directions also change with temperature. The presence of gas tends to shift the preferred orientation to higher index planes [3, Ref 52]. Bias voltage also affects the texture, e.g. in Ag, the texture changes from {111} to {200} to {111} as the voltage is increased.

(b) **Residual Stresses**
High residual stresses can cause problems by deformation and cracking of the coating or substrate or, if associated with poor adhesion, at the interface. These stresses arise from (i) atomic mismatch, and (ii) thermal expansion mismatch of the coating and substrate [3].

Residual stress due to the first cause is minimised by an increase in deposition temperature; that due to the second cause is minimised by having similar thermal expansion coefficients and thinner coatings ($<$ 1 micrometre) [6]. Bias voltage can also be used to change residual stresses. For example, a maximum compressive stress has been found [8] in Cr-sputtered deposits at a bias voltage of approximately -300V. Graded interfaces formed by changing other deposition conditions or materials, and by diffusion, can minimise the second type of residual stress.

Defects in evaporated coatings can be caused by small droplets which arrive at the substrate and are poorly bound to the coating. In the case of an alloy, the droplet can be of different composition.

Pits are formed if the droplets subsequently become detached or, if they remain on the surface, they can act as areas of preferential growth and form flakes [3].

3.2 ION PLATING

Ion plated coatings show similar morphology changes to those previously described for evaporated and sputtered coatings when the energy of the incident ions is increased (though not necessarily in the same temperature regions) [3, 32]. The fractographs shown in Figs. 12 to 15, [34], depict the different coatings formed as the plating conditions change for copper plated onto a nickel substrate. The changes are either in plating bias voltage or in gas pressure; both parameters affect the power density at the specimen. A higher bias power density causes a higher substrate temperature. The temperature of the substrate increases with bias power density in the order Fig. 12 (373K), Fig. 13 (418K), Fig. 14 (443K) to Fig. 15 (458K). The increased temperature caused by an increased gas pressure at constant bias voltage, produces a refinement in grain structure, from a coarse-grained structure with weak, poorly defined grain boundaries to an equiaxed fine-grained structure with strong grain boundaries. This effect is shown, in order of increasing pressure and temperature, in Fig. 12, Fig. 13, Fig. 15. The higher the deposition rate the coarser the structure, while a high power density refines the structure.

The porosity of ion-bombarded coatings is much less than for either sputtered or evaporated coatings. Porosity is decreased by sputter-cleaning, ion-bombardment cleaning and enhanced atomic diffusion of the coating; these processes are assisted by lower gas pressures, higher incident energy, higher substrate temperature and higher power density.

Ion-plated coatings are much more likely to have a graded-fusion zone between the coating and the substrate, though the degree to which the diffusion takes place depends very much on the energy of the ions and the substrate temperature.

3.3 ION IMPLANTATION

Because of the much higher energies involved in ion implantation compared with the other coating techniques, (accelerating voltages to 1Mev and higher) the ions penetrate much further into the substrate and therefore do not produce a coating as such, but rather a modified surface layer [11]. In most substances, the concentration distribution of ions is approximately Gaussian. This distribution depends on a wide variety of process conditions, e.g. ion energy, temperature, crystal orientation, ion type and subsequent thermal-processing [35]. Thus, to achieve a

[10]

uniform implant concentration with depth, it is necessary to make several implantations at different energies to approximate a plateau by the sum of a set of Gaussian distributions. In most systems, the depth of the implantation-induced changes is approximately 1 micrometre [11]. Fig. 16, [36], shows the changes in ion penetration that could be expected as the incident ion energies are changed for iron ions in an iron substrate. It can be seen that a constant density of implanted ions could be obtained by varying the dose for two or three selected implantation energies. The depth of implanted ions is sometimes enhanced by channelling effects which occur when the incident ions are steered into the substrate between "walls" of appropriately oriented planes of substrate atoms. The ions can therefore reach greater depths than those reached by randomly directed ions or in randomly oriented substrate planes. The proportion of channelled ions, however, is relatively small [11]. (Channelling patterns and properties are used as a technique for studying ion energy loss interactions and the substrate lattice structure [11].) The relation between the ranges of particles and their energy has been of great interest to nuclear physicists, and several theories are available for the energy loss and distribution of implanted ions [17].

Because of the high energy of the incident ions , radiation damage of the substrate is also higher for implanted ions. Upon impact, the incident ion can lose energy at the rate of 100eV/A up to 1000eV/A as it embeds in the substrate. Since the binding energy of atoms in their lattice sites is of the order of 20eV, it is to be expected that considerable radiation damage will occur. The injection of ions and formation of defects tends to promote swelling, thereby causing large compressive stresses [24]. However, in the vicinity of the surface at room temperature, even under saturation damage conditions, the fraction of atoms which are dislodged from their equilibrium lattice positions is low, usually about 10^{-3} [9].

Some of the radiation induced defects are shown schemat'cally in Fig. 17 and tabulated in Table 6. These defects may cause the effect desired by ion implantation i.e. the formation of a layer with desired properties, e.g. compressive surface stresses, hardness or chemical reactivity. Induced defects may also enhance the mobility of the impinging ions.

Unusual metastable phases may be formed in the implanted surface because of the high energy-densities of the incident ions, and the effective rapid cooling, e.g. Ta in Cu or even P in Fe, or glassy or amorphous phases if the crystallinity of the surface is reduced by heavy implantation [17]. Amorphous layers may be considered to be produced by the extremely rapid quenching of the high energy ions when they impact the surface. Amorphous layers are much more likely to be produced in non-metals e.g. Si and GaAs, than in metals. Nevertheless, glassy surfaces are possible in systems such as rare earths in Ni, or As in Fe [37]. Surfaces may be formed with chemical, magnetic, optical or mechanical properties very similar to bulk alloys of the same metals.

During ion implantation, surface atoms are also being sputtered off the surface, and a dynamic equilibrium is eventually established in which the proportion of source atoms which can be incorporated into the matrix is usually below 50% [24].

[11]

4 APPLICATIONS

In spite of the already widespread uses of vapour deposition, especially evaporation and sputtering, many commercial uses have not been fully recognised because of a lack of understanding of the properties of the surface layers . Excluding microelectronic applications, the surface properties that can be readily affected by surface modification are shown in Table 7.

The largest uses by far for the deposition techniques discussed are in the coating of Al onto plastics, in thin film microelectronics and in the optics industries [3]. Nevertheless, work is being done to examine the other potential uses of the processes. Of these processes, the most important areas for development seem to be lubrication and wear, and corrosion and oxidation resistance, all of which have a potentially enormous benefit if improvement in properties can be achieved.

4.1 LUBRICATION AND WEAR

The two main areas of surface modification are in tool and die wear and in bearings.

4.1.1 TOOL AND DIE WEAR

Hard materials are, in general, good wear-resisting materials, and elements having large atomic radii are usually oxidation-resistant. Rare earths such as La, Hf, Ru, Pt, Os, and Ir are therefore likely contenders for wear-resistant films [38]. Hard intermetallic compounds are also good for wear-resisting films and are used to improve the life of cutting tools [3]. In particular, carbides and nitrides e.g. TiN and TiC, can be sputtered onto cutting edges for improved tool life, reduced tool force and improved surface finish [3, Refs 96,97,82] and wire-drawing die life [17]. These coatings also improve the tool life of Co-cemented WC tips by a factor of up to 10. Reactive ion plating processes can be used to form TiN on cemented carbide tools [39].

Ion implantation does not seem to be widely applied commercially to dies at this stage, although nitrides, borides and carbides on many steels have been shown to reduce wear rates in tests by a factor of up to 200 [24, Ref 12, 40]. Fig. 18 shows the decrease in the pin-on-disc wear rate for steel implanted with 50keV nitrogen ions compared with unimplanted steel. The improvement is especially marked at low loads [40, 41]. Implanted nitrogen ions can also improve the wear-resistance of nitrided steel [24]. Trials on cutting and punching tools have shown that tool life could be increased by a factor of 4 with nitrogen implantation into chrome - carbon steels, tool steels and chrome-plated coatings. A twelve-fold increase in life was observed in nitrogen-implanted WC - Co knives used in abrasive wear. It has been suggested that the nitrogen distorts the Co lattice in Co-cemented WC, thereby hardening the Co binder so that the reduced wear of the binder lessens the undercutting and loss of carbide grains [24]. It is also reported that boron, nitrogen and molybdenum implanted into nitrided steel can reduce sliding wear by factors of 10 to 30 [42]. However, nitrogen implantation is not always of major benefit. As seen in Fig. 19 [43], there is no significant difference between a nitrogen (75 keV) implanted and unimplanted AISI 52100 steel in a ball on cylinder wear test. On the other hand, a titanium (150 keV) implant reduces wear by nearly an order of magnitude. The implanted titanium ions cause an excess of carbon atoms near the surface of the steel and promote the formation of TiC [43].

Tools used at high temperatures, e.g. high speed twist drills and gear cutting tools, may not show any benefit from ion implantation, possibly because of enhanced nitrogen diffusion if the surface temperature becomes too high.

A further example of reduced wear is in oil burner nozzles implanted with Ti ions. These nozzles were still as-new after 8000 hours of operation, whereas untreated nozzles were discarded after 3000 hours [42].

[12]

4.1.2 BEARINGS

Sputtering and ion plating are used to modify wear and friction characteristics, often using the same compounds or metals as are used for ion implantation [6]. Such coatings as carbides, silicides and nitrides have all been sputtered and ion placed to reduce wear, and films of MoS_2, Ag, Au, Pb have been used to provide effective lubrication. A sputtered coating of MoS_2 over Cr_3Si_2 on bearings produced a five-fold increase in life compared with a coating of only MoS_2, as shown in Fig. 20 [44, 7]. The Cr_3Si_2 is thought to coat the surface with a hard, even, glossy coating that helps prevent metal-to-metal contact and reduce dislocation movement near the surface. The effectiveness of the MoS_2 as a lubricant in reducing wear is often affected by the adhesion of the coating to the substrate, coatings tend to separate at their outer edges, with plating usually having greater adhesion than sputtering which is, in turn, more effective than evaporated coatings [12, 38]. A comparison of the effectiveness of sputtered and plated silver coatings shows that ion plating is much more effective in reducing friction than sputtering [38/Fig5].

In bearing applications, as for tool wear, ion implantation is still in the experimental stage, and studies are still being carried out to evaluate the effect of ion implantation on bearing life. Results currently show that ion implantation offers an alternative method for incorporating solid lubricant films into surfaces. A major advantage of ion implantation is that the dimensional changes that occur are very small, also there is some indication that, as the surface wears, the implanted ions move further into the substrate [24]. Other advantages of ion implantation are that the smooth transition of the source atoms into the substrate reduces the probability of spalling by decohesion and normally insoluble species can be implanted into the substrate without affecting bulk properties.

Friction tests have shown that Mo, Sn, Pb, In, Ag implanted into steel will reduce friction coefficients by up to 50% as seen in Fig. 21 for En352 steel implanted with 380keV tin ions [41]. Also, a single dose of Mo and twice that dose of sulphur ions reduces the coefficient even further, though the presence of MoS_2 molecules has not yet been proven. W and Se co-implantation has also been shown to reduce friction coefficients. It should be noted, however, that friction can also be increased by implantation. Other combinations include SbO_2 and, for higher temperature applications, PbO, B_2O_3, CaF_2 and BaF_2 on silicon nitride. The effect of ion implantation on friction is affected by the presence of other lubricants, with tin having most effect under non-lubricated conditions and Mo and S being most effective under lubricated conditions [24]. The effect of various implanted species depends on the property governing the wear mechanism, e.g. high hardness (N,C), oxidation resistance (Au) or appropriate crystallinity (MoS_2) [12].

An example of more complicated surface treatment is found in research on bearings at N.R.L., which indicates there may be some benefit if steel bearing surfaces are hardened by implantation with nitrogen or carbon, codeposited with Mo and S or W and Se and then subsequently heat-treated to form MoS_2 or WSe_2 precipitates.

4.2 FRETTING

Fretting occurs by complicated wear mechanisms involving adhesion, oxidation,abrasion and fatigue. The reduction of any of these processes is likely to prove beneficial in reducing fretting. With appropriate deposits, all deposition techniques have been shown to be effective in reducing fretting. The effectiveness of the technique seems to depend heavily on adhesion of the coating, e.g. Au films deposited onto steel by ion plating had a life 10^4 times that of Au deposited by evaporation [38]. Ag and other easily oxidised metal films have poor durability compared with oxidation-resistant films. Ion-plated boron carbide shows very good ability to reduce fretting; Cr, Au and Cd coatings are also very effective.

Results of fretting tests for gold-coating on mild steel are shown in Fig. 22 [12], for various coating processes. It is seen that the number of cycles to disruption under fretting fatigue conditions is increased by a factor of 100 for sputtering compared with evaporated coatings and a further factor of between 2 and 10 for ion plating, depending on plating conditions [38].

The effect of C, N and B on the fretting properties of Ti has been studied [2]. Boron, surface-diffused into Ti, produces a hard, thin, stable film that reduces sliding wear at certain loads by up to 1000, and markedly increases the transition load at which metallic wear supersedes normal oxidative wear [45].

4.3 FATIGUE

Fatigue life is sensitive to surface condition through the influence of time to crack initiation. Thus, modification of surface properties, e.g. by introduction of compressive residual stresses, by changing surface chemistry, or by changing surface alloy composition, can affect fatigue properties [24, 46]. The mechanisms by which ion plating and implantation affect fatigue properties is uncertain. In some cases it appears that compressive stresses are important although in other cases these stresses do not appear to be effective in increasing life to failure.

The effect of ion plating on fatigue life is being studied [2, Ref 27], with conflicting results. Al plated onto steel marginally improves the fatigue life compared with unplated steel, [47], whereas 1 micrometre of Pt ion-plated onto Ti alloy (Ti-6Al-2Sn-4Zr-2Mo) F100 engine compressor blades will increase their high temperature fatigue strength by up to 30% and high temperature fatigue life by up to fourteen times, Table 8 [49, 2, 48]. It has been suggested that the mechanical properties of the Ti alloys are improved by the suppression of oxygen-diffusion into the Ti and by giving higher creep-resistance[2, 48], and also by the plating causing compressive hardening of the alloy surface [49].

Ion-plated gold films improve the fatigue lives of steel by up to three times and the fatigue limit stress by approximately 10%, and by up to 35% if annealed subsequently to plating Fig. 23 [38], possibly because of the greater diffusion and bonding at the interface [38]. Iron-plated coatings, however produce no increase in fatigue limit, thus indicating that a coating by itself is not sufficient to decrease crack initiation [38]. Similar increases of up to 30% in fatigue lives of nickel specimens are found following a plating of Cu or Au, [15]; it is suggested this increase is caused by the surface solid-solution strengthening effects induced by the plating.

Little work has been done on the effect of ion implantation on fatigue life. Apart from the reduction in fretting-induced fatigue, ion implantation also shows evidence that nitrogen, implanted to 2×10^{17} atoms per cm^2 at 150keV, will increase the fatigue life of a rotating AISI 1080 steel beam by up to 100 times [50]. Similarly, a ten-fold improvement in fatigue life of nitrogen-implanted Ti, stainless steel and maraging steels has been reported [17, Ref 11].

Nitrogen and carbon implantation (75keV) into Ti-6Al-4V is reported to increase the fatigue endurance limit from 500 to 600 MPa (72 to 87 ksi), Fig. 24 [51, 52], possibly by the formation of titanium carbides and nitrides. Neon ions were not effective in affecting fatigue properties even though they caused compressive surface stresses [53]. Similarly, for a constant stress amplitude on Ti-6Al-4V, the effect on fatigue life is seen to depend on the dose of carbon ions (75keV) implanted and can be increased by a factor of 5 after a small dose, Fig. 25 [51].

A possible explanation for the improvement of fatigue life by implantation is that the mobility of surface dislocations is reduced, and thus slip step formation and crack initiation is suppressed [17, 24]. Compressive surface stresses due to the implanted ions might also increase the time to initiation, though as previously mentioned, these stresses are not always effective. Much further work needs to be done to investigate the effects of ion implantation on fatigue life.

[14]

4.4 HARDENING

As described earlier, ion implantation can significantly increase surface hardness, and thereby affect related properties such as wear and fatigue [2]. Much of the work has been done on iron and steel, implanted with N, B, C [40]. The effect of ion implantation on strength depends on the way the effect is measured, e.g. whether total bulk properties (as in tensile tests) or surface properties (as with indentation microhardness) are determined. An example of the latter is shown by doubling of the microhardness of beryllium implanted with boron. The microhardness is even higher after a post-implantation anneal [40]. Most of the hardening can be attributed to formation of nitrides, borides, carbides and radiation defects [40]. The inert gases Ar and Ne have no effect at higher temperatures, but do harden at lower temperatures. Yield and tensile strengths of stainless steel wires have been increased slightly by ion plating with B_4C, Cu and Ti, Fig. 26 [38]. The increase is possibly caused by the reduction in surface slip and step formation [38], especially after the various post-plating heat-treatments, whereas a very hard material such as tungsten shows very little effect from ion plating since the small effect of surface-hardening is masked by the already high bulk strength, Fig. 27 [38].

4.5 CORROSION

All of the deposition techniques have been widely exploited for increased corrosion and oxidation resistance through the modification of chemical properties of the surface. The techniques vary from simple surface-coatings (e.g. by evaporation and sputtering), to prevent environmental degradation of the surface of bulk components, to surface-alloy modification by ion implantation.

A major benefit of surface coatings can be the reduction in the use of strategic and expensive metals such as Pt, Cr and Co due to the small quantities of these metals required to produce high concentrations in the surface layers only.

Two factors that seem to be most important are the nobleness of the implanted metal and its ability to form durable protective films. The incorporation of a more noble metal leads to improved corrosion-resistance, while elements such as Cr, Ni, Ti, Al are successful because of their stable adhering oxides [17].

The probability of film failure by simple oxidation, by thermally induced oxide-spalling and resulting pitting, or by crevice corrosion, is reduced by better adherence and lower porosity [54]. Ion plating and ion implantation are most successful in this regard, although poor plating conditions and techniques or subsequent abrasion, can cause incomplete coating, cracking or scratching of coated layers. Such defects then allow the ingress of corrosive agents resulting in attack of the substrate and consequential lifting of the coating. Under these circumstances, conventional cadmium-platings can offer better protection for steels than aluminium coatings [55]. Ion implantation has an added advantage compared with ion plating in that there is no definite coating interface that can be easily ruptured, and this feature is reponsible for enhanced corrosion resistance of implanted layers.

Evaporative coatings are used in the aircraft industry to coat Ni-base alloy blades used in the hot end of turbine engines, particularly to reduce hot salt effects [3, 54]. Such coatings as the quaternary M – Cr – Al – Y, (where M is any of Co, Ni, Fe) are used usually to a thickness of 20 to 100 micrometres [54]. MCrAlY coatings are designed to produce protective scales of either Al_2O_3 or Cr_2O_3 on heating in air. These coatings are relatively ductile, but increasing ductility is gained at the expense of oxidation and hot corrosion resistance, which is dependent primarily on aluminium content [35]. The yttrium is included for enhanced oxide adherence, and chromium increases the activity of Al_2O_3 formation [35].

These coatings have the advantage over thermal diffusion coatings that they cause minimal loss of mechanical properties. A disadvantage is spallation of the Al_2O_3 layer caused by thermal cycling. An example of the

effectiveness of CoCrAlY plating on a steel substrate in reducing corrosion is shown in Fig. 28 [56]. It is seen that the corrosive metal loss from the metal surface in a simulated turbine environment, due to sodium in the fuel/air mixture, is reduced by a factor of approximately 10 after ion plating (4.5keV).

Modified SiC and other carbides have been sputtered on engine components such as compressor blades to reduce oxidation and erosion [6, Ref 8].

Continuing attempts to increase the effectiveness of coatings on gas turbine engines has led to superior properties of ion-plated coatings over evaporated and sputtered coatings. Ti alloy engine components have shown marked improvement in corrosion resistance after ion plating with 1 micrometre films of Pt, Pt/Rh and Au [48]. The threshold stress for cracking in hot salt corrosion is also raised above 350 MPa (50ksi) by Pt and Pt/Rh whereas stress-corrosion cracking occurred even with Au plating. An advantage of Pt is its negligible solubility in Ti up to 600 degrees C, and the ion plating technique eliminates the risk of hydrogen charging the Ti during coating [49]. While Zn and Cd coatings are generally found to be most effective for protecting steel, widespread use in the aircraft industry is made of Al ion plated onto screws and bolts (McDonnell -IVADIZE process) [47,55,57,58,59]. This technique is particularly useful instead of Cd plating when Ti parts are in contact or nearby. In certain circumstances, such as in countersunk holes, Al ion-plated coatings generally outperform cadmium in inhibiting corrosion in SO_2 spray tests

[47]. Further applications of aluminium plating are made by McDonnell Aircraft Company. Ion-plated aluminium is used on Harrier aircraft and will be the primary corrosion protective coating on the F-18 Hornet. On the Hornet, it will be used on all fatigue-critical aluminium structures, all high strength steel structures and on titanium and alloy steel fasteners. Large structural and engine parts that are ion plated include: landing gear, engine mounts, stator vane assemblies, aluminium wing skin. Some stainless steel parts have now been changed to aluminium-plated alloy steel [60].

Aluminium is also electron beam evaporated onto steel strip to a thickness of 6 micrometres to replace tinplated steel; 10% of "tin" can manufacture in West Germany is Al coated steel [3]. Aluminium has also been ion plated (to 10 micrometres) onto uranium to reduce corrosion, especially under very humid conditions such as those experienced by fuel elements in some reactors [61]. The coating also withstands the thermal and mechanical shock experienced in the reactors [61]. Fig. 29 [61] shows the corrosion rates for aluminium-plated uranium for various plating conditions. It is shown that plating produces significant reductions in the rate of corrosion. Higher ion energies and heat treatment to allow diffusion at the U-Al interface, also decrease the corrosion rate [61].

A characteristic of ion implantation is the formation of alloys, including metastable alloys, at the surface without the heat treatment enhanced diffusion of other techniques. In several instances, ion implantation of the above metals onto a steel substrate has provided the same corrosion and oxidation resistance as would be obtained from a bulk alloy with identical composition to the surface composition, although pitting and corrosion resistance may not be as good [26]. Such alloys as Fe-Cr and Fe-Ni have been formed with doses of 25 kV ions to a dose of 4×10^{16} ions per cm^2 [26]. Fig. 30 [26] shows the oxide thickness for specimens oxidised in air at $320^{\circ}C$. It is seen that an iron surface implanted with chromium to an effective surface composition of Fe-24Cr has similar oxide thickness to an Fe-18Cr bulk alloy. Both systems show much slower oxide formation than unalloyed iron [26]. Turbojet engine bearing steel (AISI-M50) has its corrosion-resistance greatly enhanced by Cr and Mo implantation, especially if Cr and Mo are co-implanted [62].

Similarly, rare-earth ions, e.g. yttrium, implanted into steel improve high temperature oxidation of Cr-alloyed steels without the disadvantage of degraded ductility and tensile strength (due to grain boundary segregation) accompanying bulk-alloying with rare-earths [17, Refs 18,19,20,40]. The corrosion properties of Y or La implanted steel compared favourably with those of a corresponding stainless steel of 0.4 wt% Y added throughout the bulk [1, 63], possibly due to the formation of impermeable

[16]

barriers of $YCrO_3$ or $LaCrO_3$. Yttrium has halved the oxidation rate of steel in a CO_2 atmosphere, Fig. 31 [1], and the plated alloy performs as well in this respect as the bulk Y-steel alloy. A further coating of Al can give additional protection to rare-earth-protected steels. The effect of the rare-earth metals is to form a stable oxide at the surface, or to form stable perovskites such as $CaTiO_3$ or $EuTiO_3$, and thus prevent further diffusion of oxygen into the substrate. Similar protection is obtained with rare-earth and alkaline-earth-metals e.g. Ca, Ba, Eu implanted into Ti [24]. Implantation of Ti with Pd to a concentration of 5at%, 250A below the surface, leads to higher resistance to attack by H_2SO_4 [64, 7]. Even though the Pd ions are attacked and removed, the Ti metal is removed even faster, with the result that the Pd concentration at the exposed surface rises to 20at% and the attack rate drops by up to 1000. There is also evidence that ions, e.g. Cs, Ba, Ru, that lie along diffusion paths of the corrodent, e.g. grain boundaries and dislocations, are more effective in reducing corrosion. A 76% reduction in oxidation rate of Ti has been achieved by implanting Ca, and a 52% reduction in stainless steel by implanting Bi ions [42].

4.6 OTHER APPLICATIONS

The various deposition techniques are also used in other areas of application viz.:

4.6.1 CATALYSIS

High surface areas can be readily formed by coating a relatively thin layer of expensive catalyst on a less expensive substrate [3].

4.6.2 ADHESION

The effect of bombarding surfaces with high energy ions is to remove surface layers by sputtering, and leave an atomically clean surface [27, 65]. Coatings applied by evaporation, sputtering or ion plating, either during or after this cleaning have shown enhanced adhesion. One example is the improved adhesion of vacuum-evaporated films on glass cleaned by ion bombardment, Fig. 8 [27]. Ion implantation improves the adhesion properties of metals on glass by two orders of magnitude by modifying the interface with implanted species (Ar ions at 10^{16} ions per cm^2 and 120 kV) [27].

"Strike" layers can also be plated onto a surface to increase adhesion of subsequent plated layers, e.g. a plated titanium layer enabled TiC to be plated onto Ti-6Al-4V [66].

4.6.3 OPTICS

Ion-beam polished surfaces are equal to the very best mechanically polished surfaces with only 0.06% of the light being scattered. Laser mirrors can also be polished using sputter-cleaning [11].

Areas in which ion implantation is being investigated for optics applications include the use of ion implantation to reduce the tarnishing of metal mirrors and to improve their mechanical properties. Ion implantation might also be able to make filters and windows more selective by controlled ion doping [17]. Ion implantation is also being investigated for the manufacture of complete optical circuits in an integrated optics system in which information is carried in light pipes with integrated switching and detection. The effect of scattering at the surface scratches and blemishes on light pipes can be overcome by implantation. Implantation of hydrogen ions into silica causes a subsurface layer of altered refractive index below the surface flaws [67].

[17]

4.6.4 SUPERCONDUCTORS

Thin film superconductors of metastable alloys can be laid onto copper substrates. Some alloys that are difficult to fabricate (e.g. Nb_3Sn) can also be manufactured using deposition techniques. These newer alloys often have higher current-carrying capacity in high magnetic fields and at higher temperatures [3].

4.6.5 BIOMEDICINE

A major concern in bio-implants is the bonding between bone growth and the metal implant [3]. Controlled porosity of the metal surface can be achieved using sputtering and plating techniques. Another application is the sputter-coating of cattle bone onto hip prostheses to promote bone growth and attachment to living bone [6, Ref9]. Corrosion resistance of hip-joint prostheses is improved by 5 times after a process in which tin ions are implanted into the titanium-alloy prosthesis and then diffused further into the surface by nitrogen ion bombardment (radiation-enhanced-diffusion) [68].

4.6.6 PLATING AND JOINING

Ion plating and implantation have been used to form a "strike" layer prior to electroplating [8, Ref28] if the substrate material is not readily plated directly. Surface coatings might also allow common joining procedures such as soldering and brazing to be used on systems that might not otherwise be joined by these processes.

[18]

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TABLES

Table 1   Some typical surface modification techniques.

PAINTING
CARBURISING
NITRIDING
ANODISING
CONVERSION COATING
ELECTROPLATING
ELECTROLESS PLATING
CHEMICAL VAPOUR DEPOSITION
PLASMA SPRAYING
ARC SPRAYING
DETONATION GUN SPRAYING
WIRE EXPLOSION SPRAYING
EVAPORATION
SPUTTERING
ION PLATING
ION IMPLANTATION

Table 2   Directions of recent work on new methods of surface
protection and coating [2]. Synopsis of 1040 citations in Metals
Abstracts Index (1970-1979).

SYSTEMS

ALUMINIUM ALLOYS	355 Citations
NICKEL ALLOYS	240 Citations
STEELS	210 Citations

NEW PROCESSES

VAPOUR DEPOSITION	689 Citations
ION PLATING	141 Citations
ION IMPLANTATION	131 Citations
RAPID QUENCH OF SURFACES	47 Citations
LASER PROCESSING	33 Citations

[23]

Table 3   Typical operating conditions and materials for the P.V.D. processes.

	EVAPORATION	SPUTTERING	ION PLATING	ION IMPLANTATION
SOURCE	HEATED RADIATION EDDY CURRENTS LASER ELECTRON BEAM ELECTRIC ARC FILAMENT HEATED	COLD GAS DISCHARGE Knocks on atoms from Target which condense on substrate.	HEATED EVAPORATED as for evaporation, and atoms ionised in discharge.	HEATED IONISED ATOMS ELECTRIC ARC PLASMA ARC DISSOCIATION LASER PENNING DISCHARGE
GAS	VACUUM	ARGON XENON	ARGON	VACUUM
PRESSURE	$10^{-3}-10^{-4}$ Pa $10^{-5}-10^{-6}$ mm Hg	3-20Pa $20-150\times10^{-3}$ mm Hg	0.5-30Pa $5-200\times10^{-3}$ mm Hg	$10^{-3}-1.5\times10^{-9}$ Pa $8\times10^{-6}-10^{-11}$ mm Hg
PATH	LINE OF SIGHT COLLISIONLESS POOR THROWING POWER	GAS COLLISION SCATTERING. GOOD THROWING POWER	GAS GLOW DISCHARGE SPUTTER CLEANING OF SUBSTRATE. GOOD THROWING POWER	LINE OF SIGHT POOR THROWING POWER
PARTICLE ENERGY	0.2 to 1eV	0.1 to 100eV	2 to 5keV	10 to 1000keV
SUBSTRATE POTENTIAL	GROUND		-2 to 5keV	10 to 1MV

Table 3 (cont)

DEPOSITION RATE	RELATIVELY HIGH 10 to 4500 μm/hr	0.25 to 250 μm/hr	5 to 1500 μm/hr	to 1 μm/hr
ADHESION	FAIR TO GOOD depending on substrate temperature.	GOOD EXCELLENT with sputter clean.	EXCELLENT with interdiffusion.	EXCELLENT
SUBSTRATE	CONDUCTING NON-CONDUCTING NON-GASSING METAL CERAMIC PAPER GLASS	CONDUCTING NON-CONDUCTING NON-GASSING NON-METAL METAL PLASTIC PAPER GLASS	CONDUCTING MUST WITHSTAND ION-HEATING. (Can be water cooled)	ANY SOLID MATERIAL SEMICONDUCTOR METAL INSULATOR
COATING	METAL ALLOYS COMPOUNDS	METAL (Especially High melting point, high conductivity metals that are not easily evaporated e.g. Pt, Pd, Au, Cu) NON-METAL INORGANIC COMPOUNDS BONE ALLOYS COMPOUNDS	METAL ALLOYS COMPOUNDS CERAMIC ANY EVAPORABLE MATERIAL	ALL ELEMENTS ALLOYS COMPOUNDS

Table 4 Typical operating conditions and economic factors for ion implantation [36]. These ranges represent typical rather than absolute limits.

Ion Species	Any element or combination
Target Material	Any solid material
Target Preparation	Surface clean and smooth
Target Dimension	Less than 2 metre
Target Temperature	$-196^{\circ}C$ to $300^{\circ}C$
Pressure in Beam Tubes	10^{-5}torr to 10^{-7}torr
Potential on Terminal	10kV to 1000kV
Depth of Implanted Ions	10nm to $1\mu m$
Ion Beam Current	1nA to 20mA
Ion Beam Cross Section	$0.1cm^2$ to $1cm^2$
Ion Fluence or Dose	10^{16} atoms/cm^2 to 10^{18}atoms/cm^2
Relative Concentration	1at.% to 50at.%
Surface erosion	1nm to 100nm
Capital Investment	$200,000 to $400,000
Power requirement	5kVA to 20kVA
Floor space Requirement	$10m^2$ to $20m^2$
Implantation Costs	1cent to $1/$cm^2$

Table 5 Typical changes in microstructure and material properties as operating conditions are changed for evaporation and sputtering.

VARIATION OF COATING MORPHOLOGY WITH PROCESS PARAMETERS

PROCESS PARAMETER VARIATION

INCREASING PARTICLE ENERGY
DECREASING GAS PRESSURE
INCREASING DIFFUSION RATE
INCREASING SURFACE-ATOM MOBILITY
INCREASING SUBSTRATE TEMPERATURE

$T_m < 0.3\,T_m$ ⟶ $0.3\,T_m < T < 0.5\,T_m$ ⟶ $T < 0.5\,T_m$
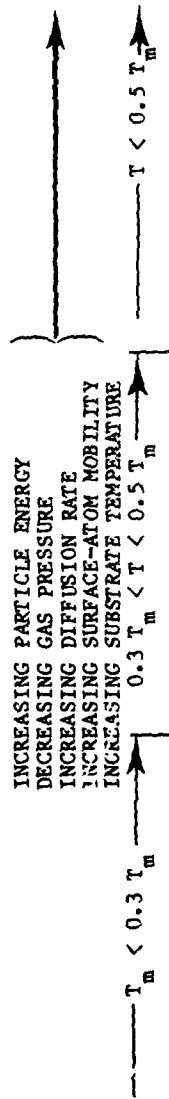
COATING MORPHOLOGY VARIATION

INCREASING STRUCTURE REFINEMENT

Small islands grow as tapered crystallites	Elongated columnar grains Definite crystallography Definite grain boundaries DECREASING DISLOCATION DENSITY	Fine, Equiaxed grains
High dislocation density. High Porosity	Lower dislocation density, especially near grain boundaries. INCREASING DENSITY INCREASING MICROHARDNESS DECREASING RESIDUAL STRESS DECREASING PREFERRED ORIENTATION	Low dislocation density.

Table 6   Radiation induced defects occurring during ion
implantation.

CRYSTAL DEFECTS

      IMPLANTED ATOMS
      VACANCIES - INTERSTITIALS
      FRENKEL PAIR
      DISLOCATIONS
      VOIDS
      BUBBLES e.g. Ar or He
      ATOMIC DISPLACEMENT
      CROWDIONS
      THERMAL SPIKES (Very short life $\sim 10^{-11}$ secs)

EXTERNAL PRODUCTS

      SCATTERED IONS AND ATOMS
      SPUTTERED IONS, ATOMS AND MOLECULES

Table 7   Applications of the P.V.D. processes.

FRICTION, LUBRICATION AND WEAR
FRETTING
FATIGUE
HARDENING
CORROSION
ELECTROCHEMISTRY
CATALYSIS
ADHESION
OPTICS
MAGNETIC
BIOMEDICINE
JOINING
DECORATIVE

Table 8 Fatigue life of uncoated and platinum coated Ti-6Al-2Sn-4Zr-2Mo F-100 engine compressor blades [49].

SURFACE CONDITION	MAXIMUM STRESS (MPa)	No. CYCLES TO FAILURE	LIFE INCREASE
ROOM TEMPERATURE			
UNCOATED	951	6.3×10^4	
PLATINUM COATED	951	19.0×10^4	200%
455°C			
UNCOATED	730	2.9×10^4	
PLATINUM COATED	730	4.0×10^4	40%
UNCOATED	703	2.6×10^5	
PLATINUM COATED	703	32.0×10^5	1100%
UNCOATED	621	6.0×10^5	
PLATINUM COATED	621	91.0×10^5	1400%

[30]

Fig. 1   Schematic of evaporation process using electron beam heating [3].

Fig. 2   Schematic of basic sputtering process [3].
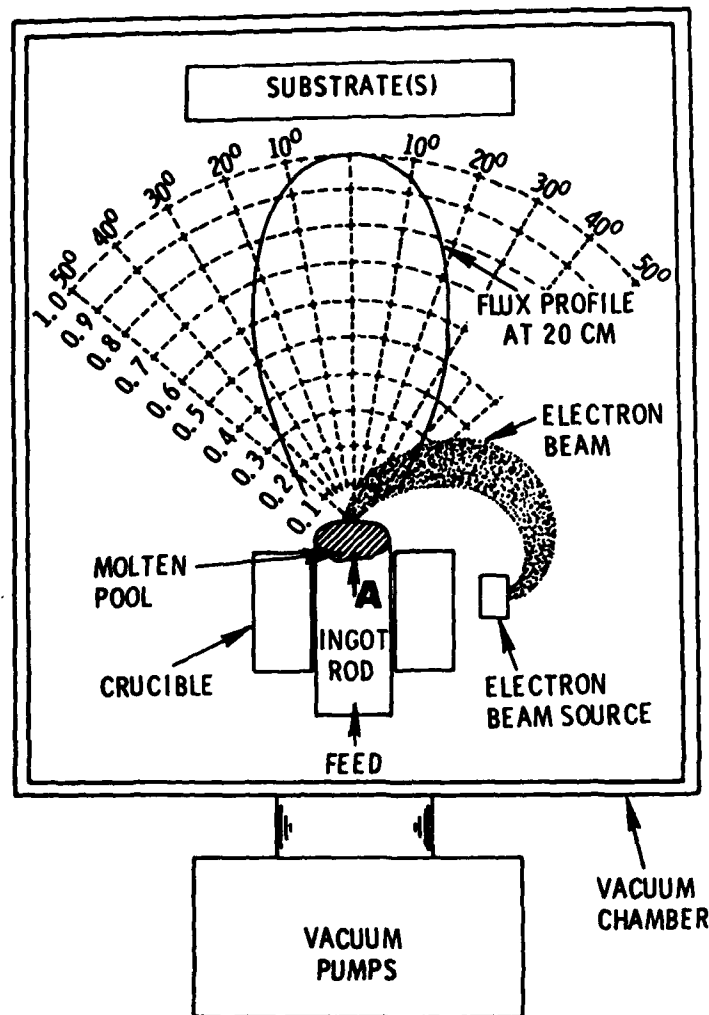
Fig. 2   Schematic of basic sputtering process [3].
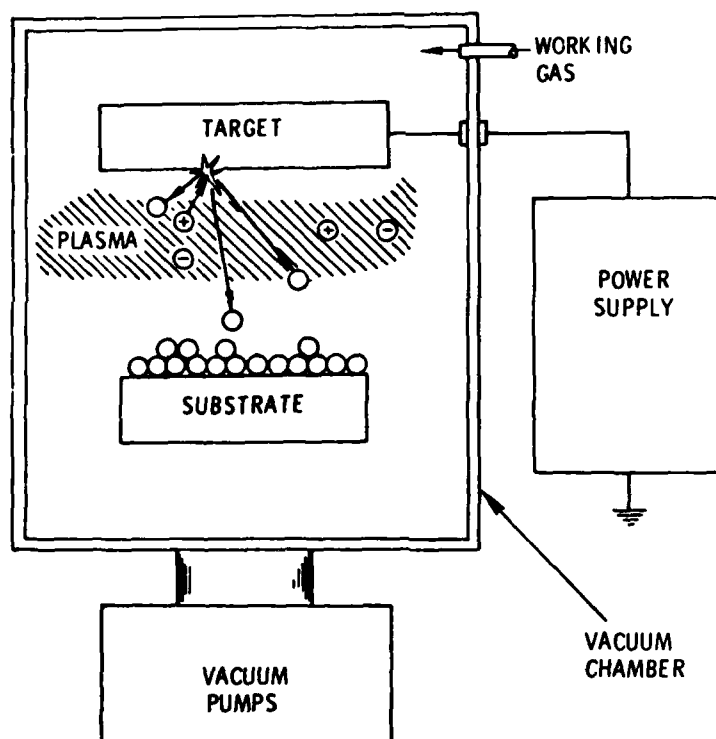
Fig. 3   Schematic of ion-plating process [3].
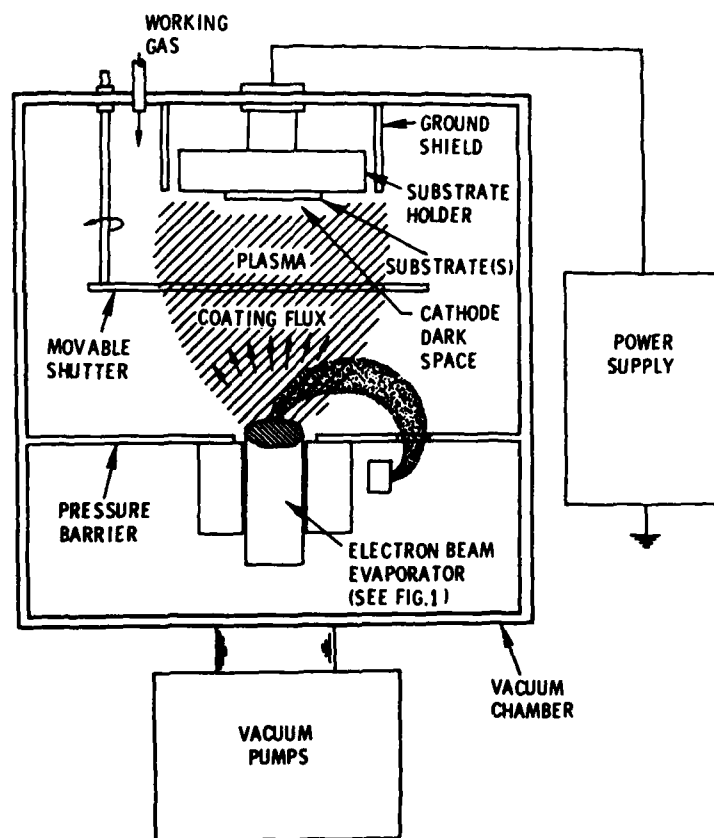
Fig. 4   Film thickness on front and back surfaces of a flat plate specimen as a function of argon pressure. These curves show the greater throwing power that occurs with greater scattering at higher gas pressures [16].
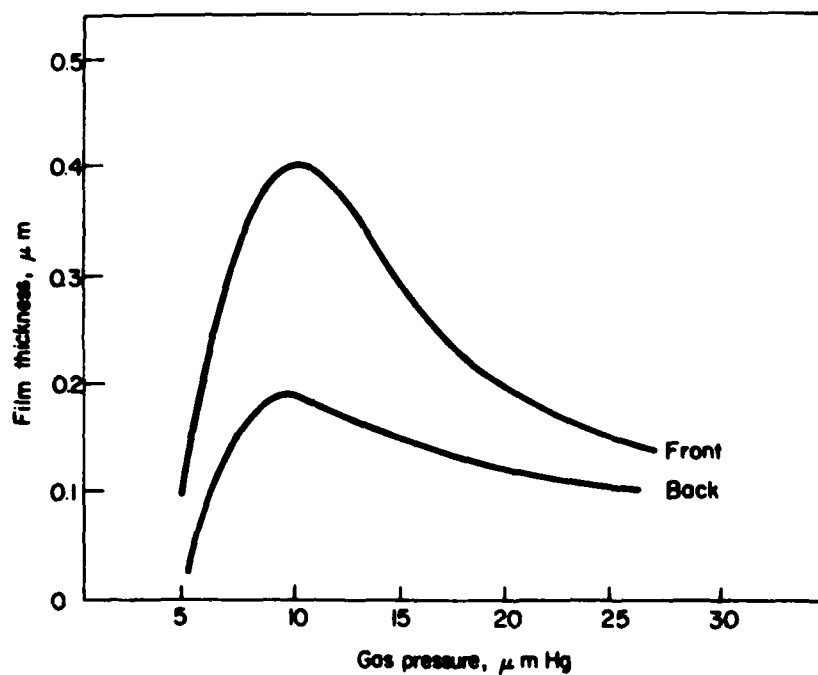
Fig. 5   Schematic of ion implantation process. Ions from the
source are accelerated to an energy of about 25KeV by the extraction
electrode and then pass through the mass analyser. The selected ions are
then accelerated to the desired energy, deflected by a scanning system and
allowed to strike the target which, in this case, is rotating to enable
uniform coating [36].
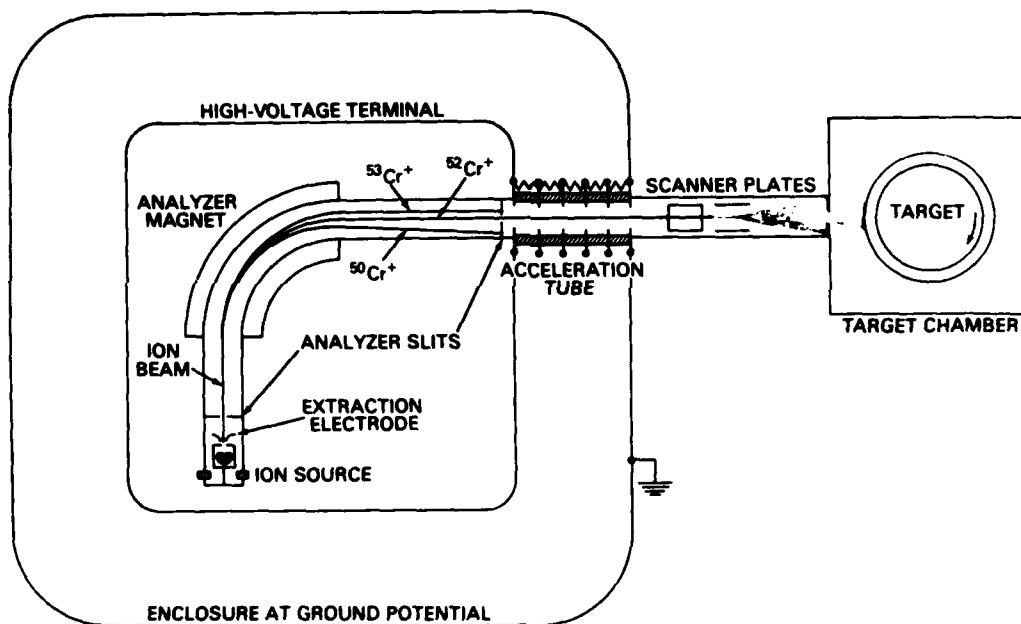
Fig. 6   Schematic representation of a number of ion paths entering
the target from the left. The resulting range curve can be either skewed to
the left or right depending on process parameters [36].
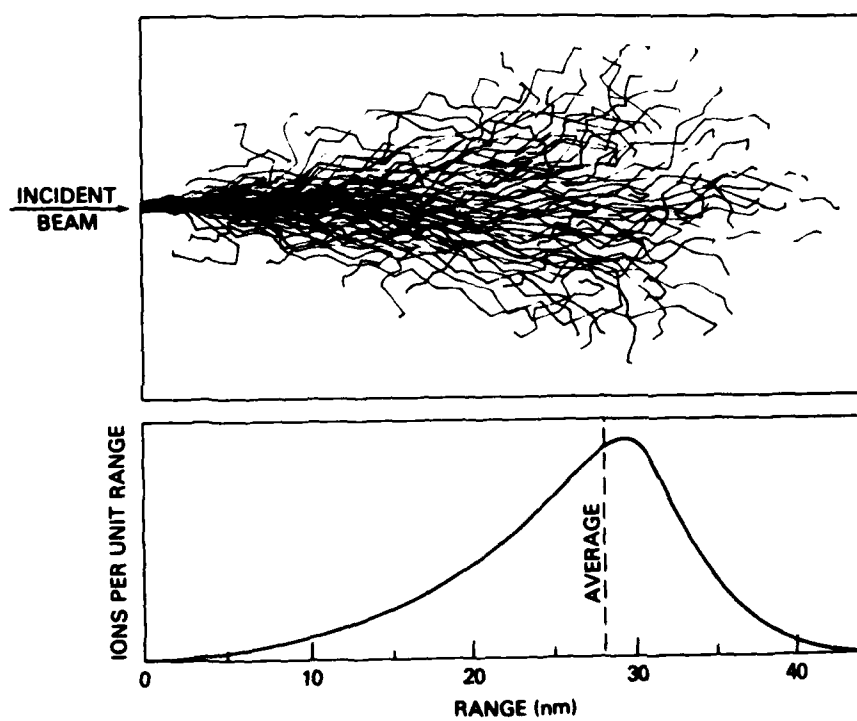
Fig. 7   Ion ranges in aluminium, showing the changes in range as the ion energy is increased and the ion type changed [11].

Ion ranges in aluminium

[37]
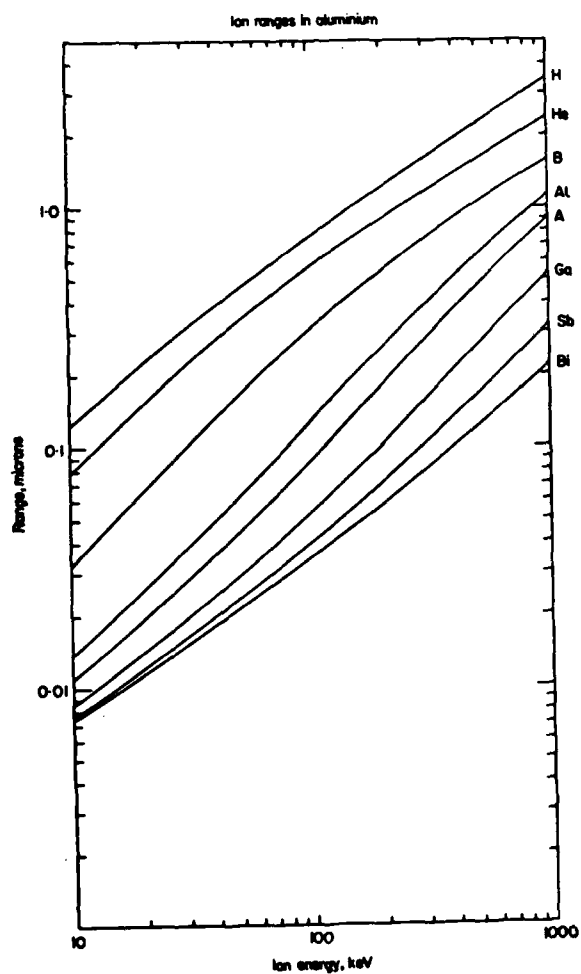
Fig. 8　　　Change in adhesion of an aluminium film to glass after ion implantation to varying doses by argon ions. The adhesion of the film is enhanced by the "stirring" of the film-glass interface. Note the araldite bond used during the test fails before the metal-glass bond after a certain dose [27].
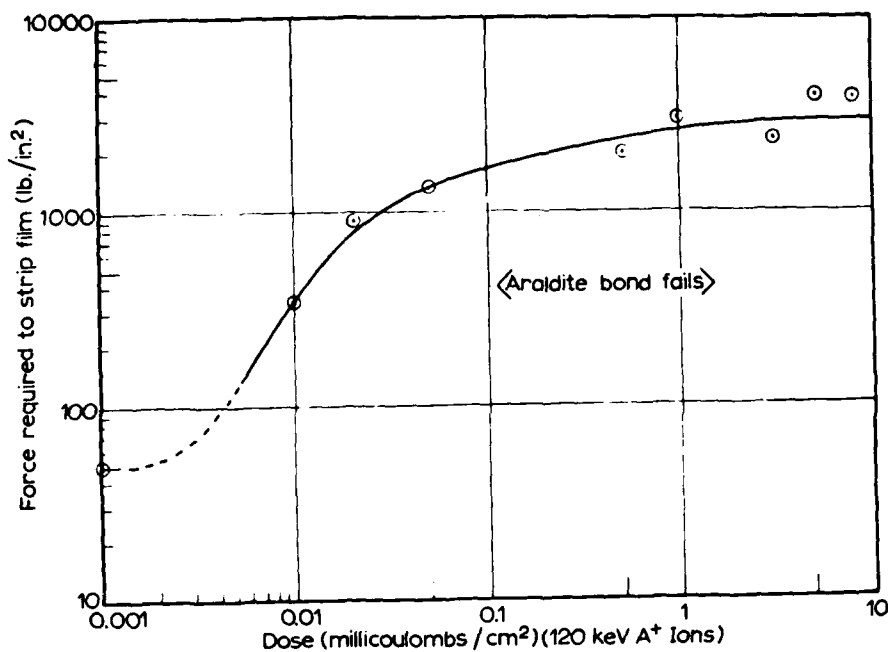
Fig. 9   Schematic   temperature   dependence   of microstructure
morphology as the temperature of the substrate is varied. T_m is the melting
point of the plated metal [30].
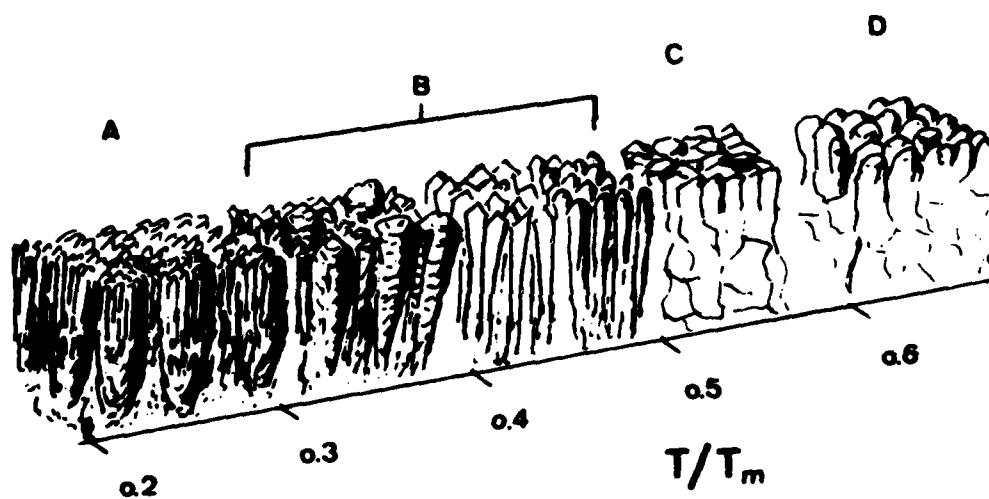
Fig. 10   Microstructure of evaporated titanium film as temperature of the substrate is varied.(a)573K $(T/T_m=0.29)$, (b)753K (0.39), (c)873K (0.45), (d)1053K (0.54) [28].
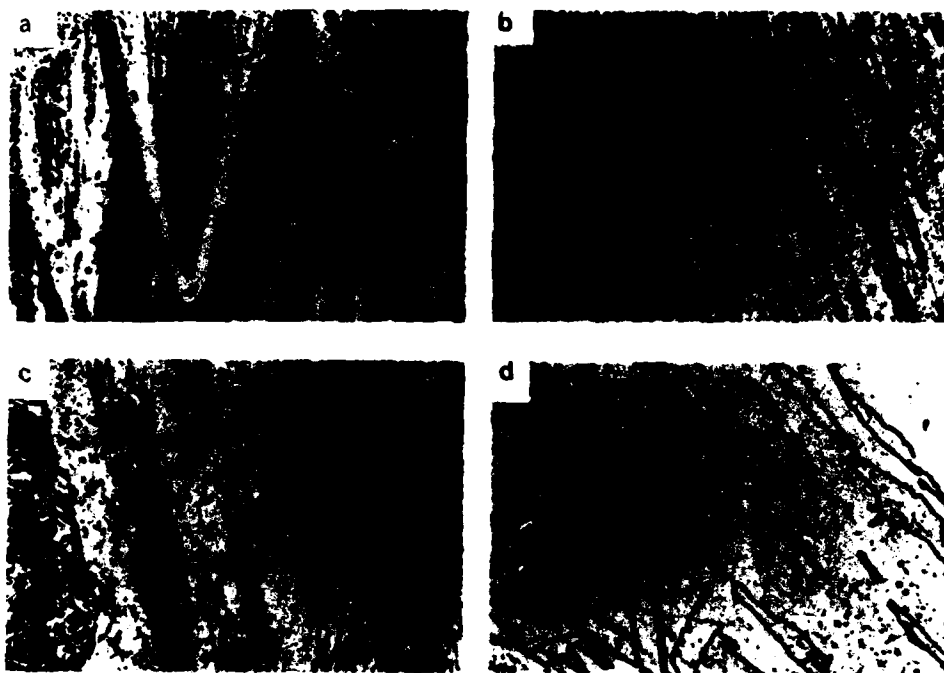
Fig. 11 Change in microstructure through cross-sections of titanium
evaporated deposits produced at various deposition temperatures [33].

900°C; 125X   860°C; 125X   650°C; 125X   570°C; 150X

500°C; 200X   460°C; 200X   315°C; 200X   275°C; 200X
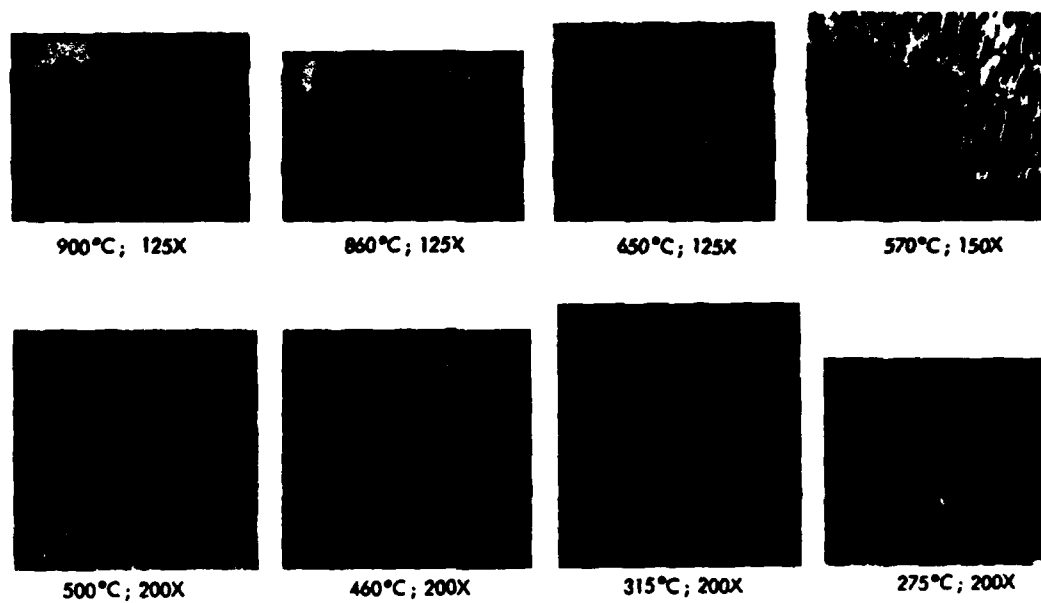
Fig. 12   Ion plated copper on nickel for 4KV bias and 1×10^{-2}mm argon pressure. Substrate temperature 373K [34].

Fig. 13   Ion plated copper on nickel for 4KV bias and $2x10^{-2}$mm argon
gas pressure. Substrate temperature 418K [34].

Fig. 14   Ion plated copper on nickel for 5KV bias and 2×10^{-2} mm argon pressure. Substrate temperature 443K [34].

Fig. 15   Ion plated copper on nickel for 4KV bias and 4×10^{-2} mm argon pressure. Substrate temperature 458K [34].

Fig. 16   Calculated ranges of iron ions in an iron substrate for
incident ion energies from 50KeV to 200Kev [36].
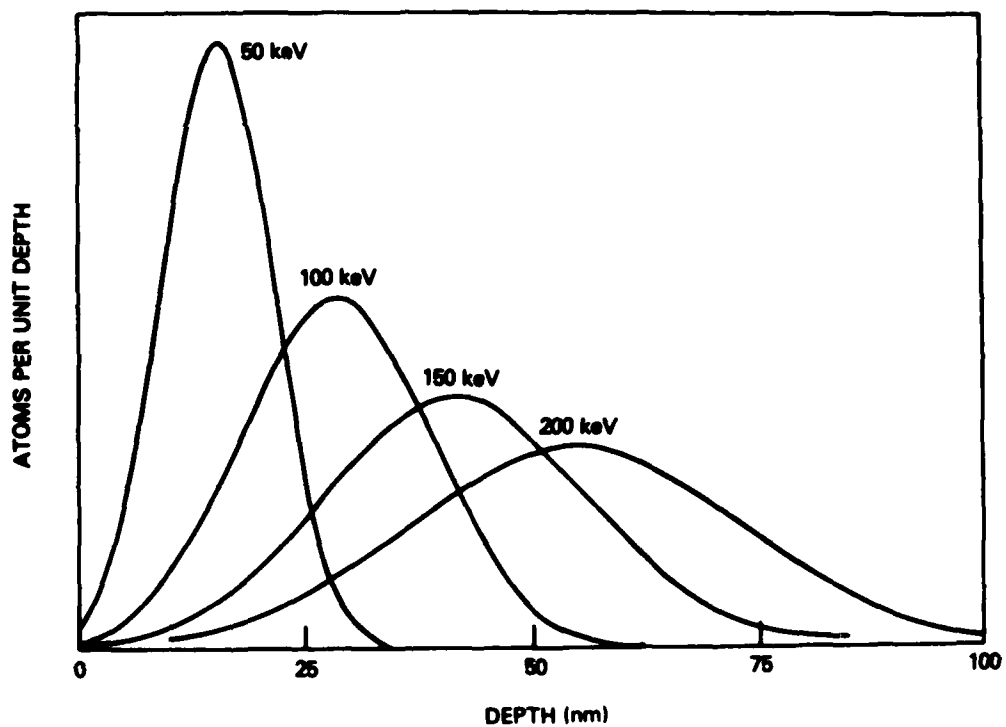
Fig. 17   *Schematic of radiation processes that might occur during ion implantation* [24].
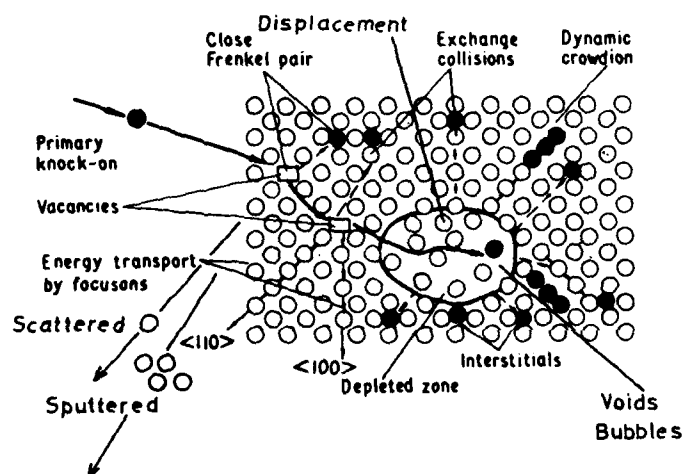
Fig. 18   Relative improvement in wear rate (i.e. decrease in wear)
as a function of dose of nitrogen ions for pin on disc tests on lubricated
En40B steel [41].

En 40 B STEEL
PIN- ON - DISC

RELATIVE DECREASE IN WEAR RATE

MEASURED NITROGEN DOSE N/cm^2 (50 KeV)
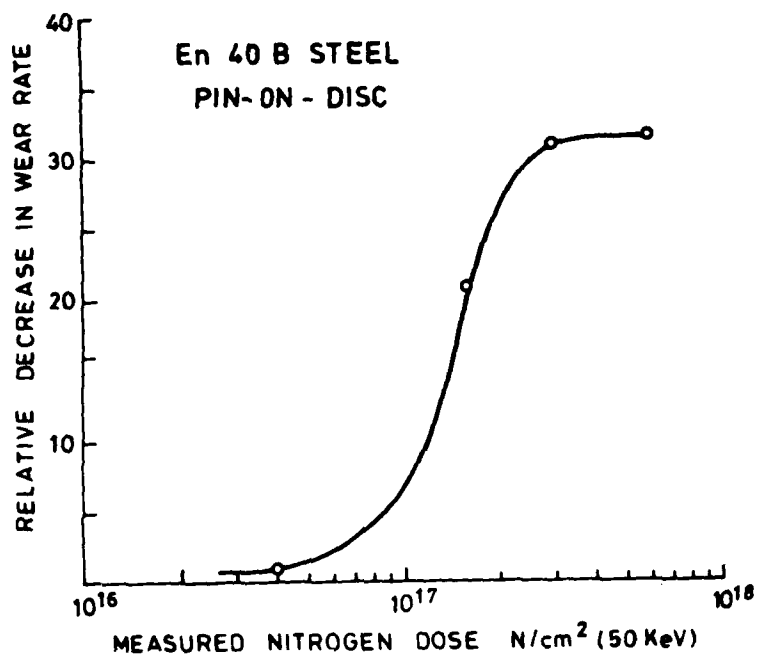
Fig. 19   Wear volume for implanted and unimplanted 52100 steel in a ball on cylinder wear test. The nitrogen implanted and the unimplanted steels show no significant difference, whereas the titanium implanted steel shows significant decrease in wear [43].
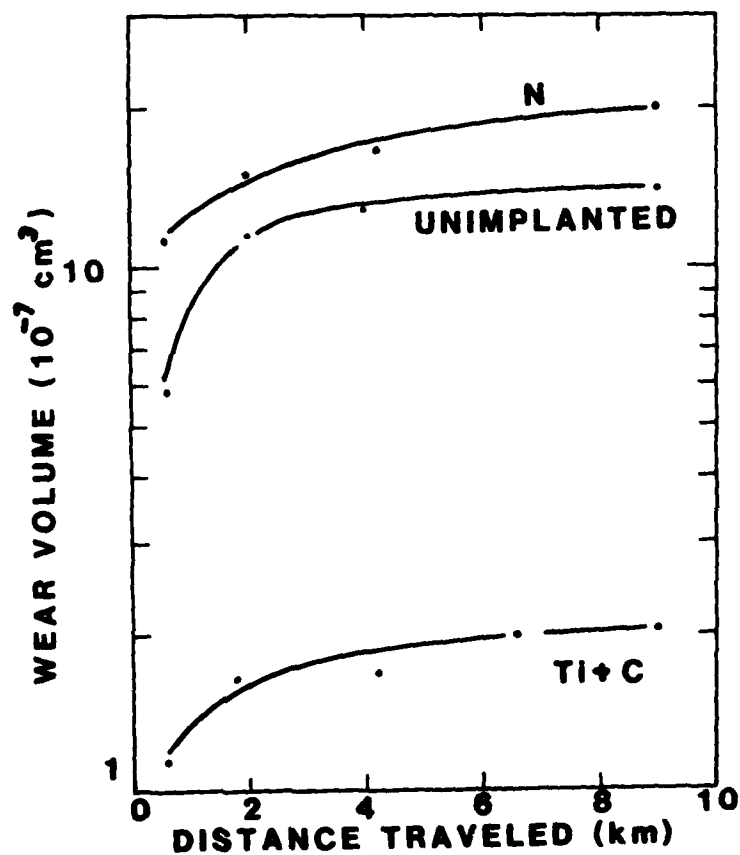
Fig. 20   Endurance lives of 440C stainless steel ball bearings with sputtered MoS_2 films on races and cage, with and without a Cr_3Si_2 underlayer [7].
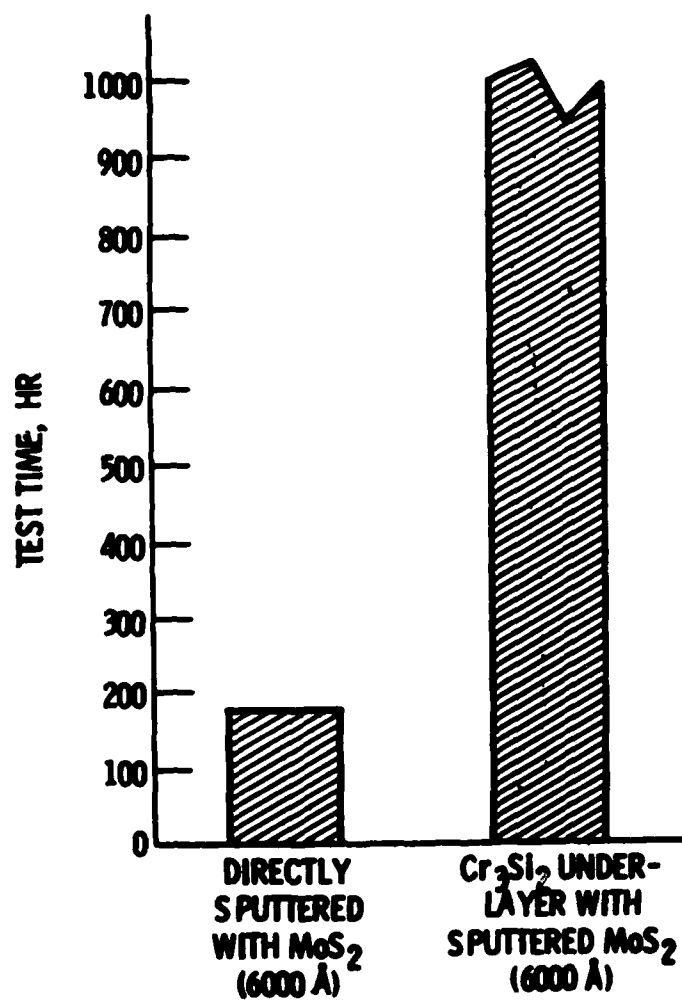
Fig. 21   Friction change measured on En352 steel ion implanted with tin. The markers show the extent of the implanted region [41].

^{120}Sn 380 keV
2.8.10^{16} ions cm^{-2}

En 352 P=2Kg
BALL DIAMETER
= 4mm.

COEFFICIENT
OF FRICTION

SURFACE TRACK LENGTH   mm.
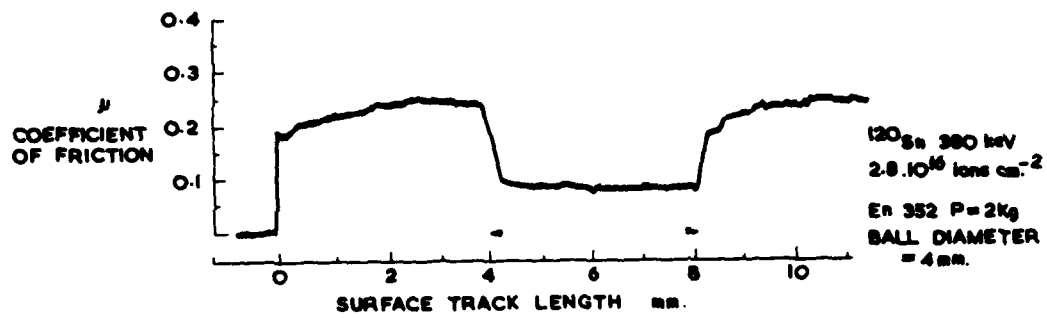
Fig. 22   Cycles to film disruption in fretting tests of gold films
on steel, showing the effectiveness of different deposition techniques
[12].
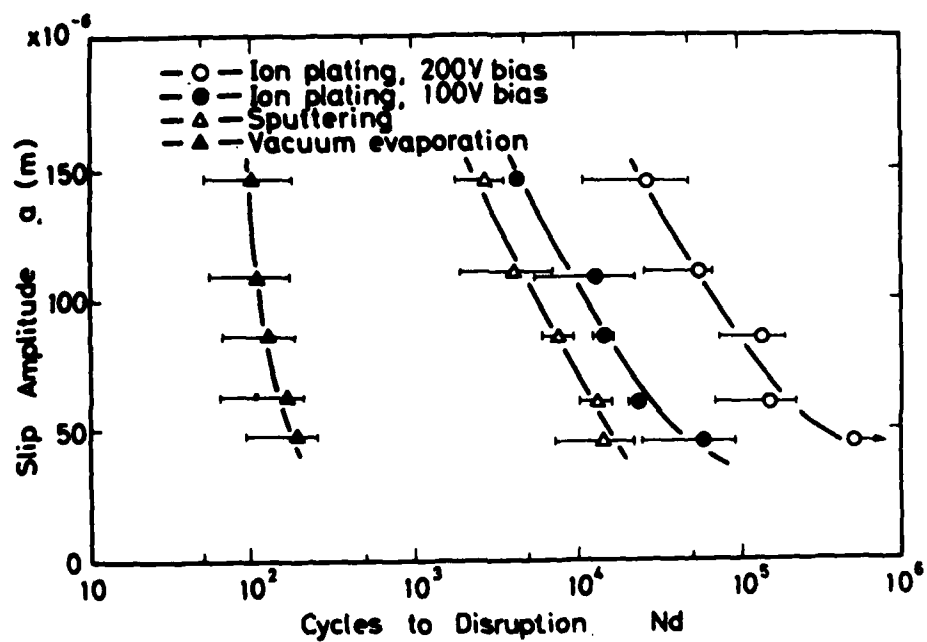
Fig. 23   Effect of ion plating on the fatigue properties of low
carbon steel after different plating techniques [38].
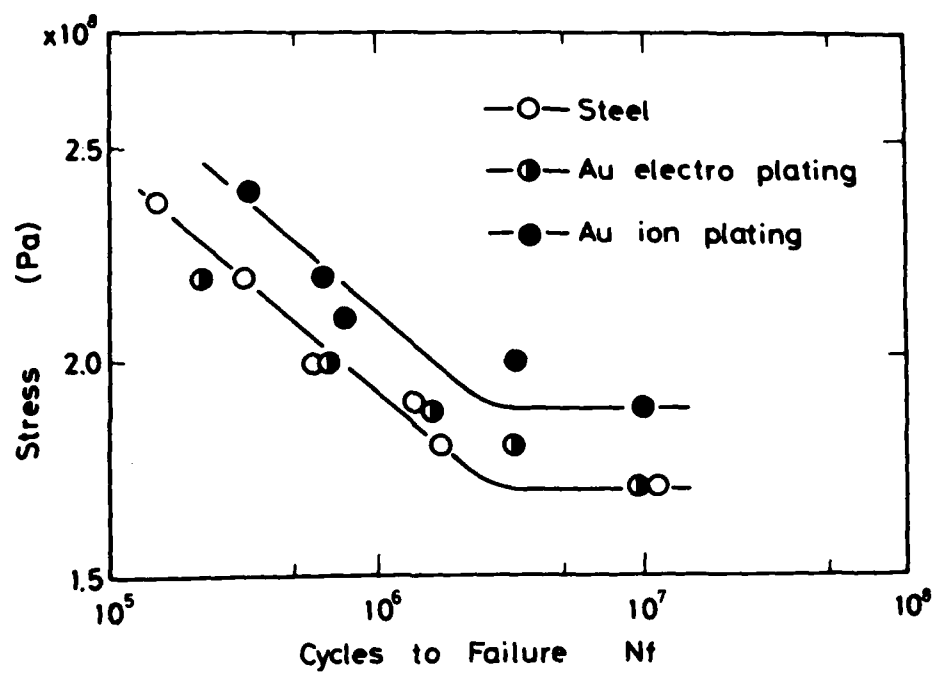
Fig. 24   Stress amplitude vs. cycles to failure for mill annealed
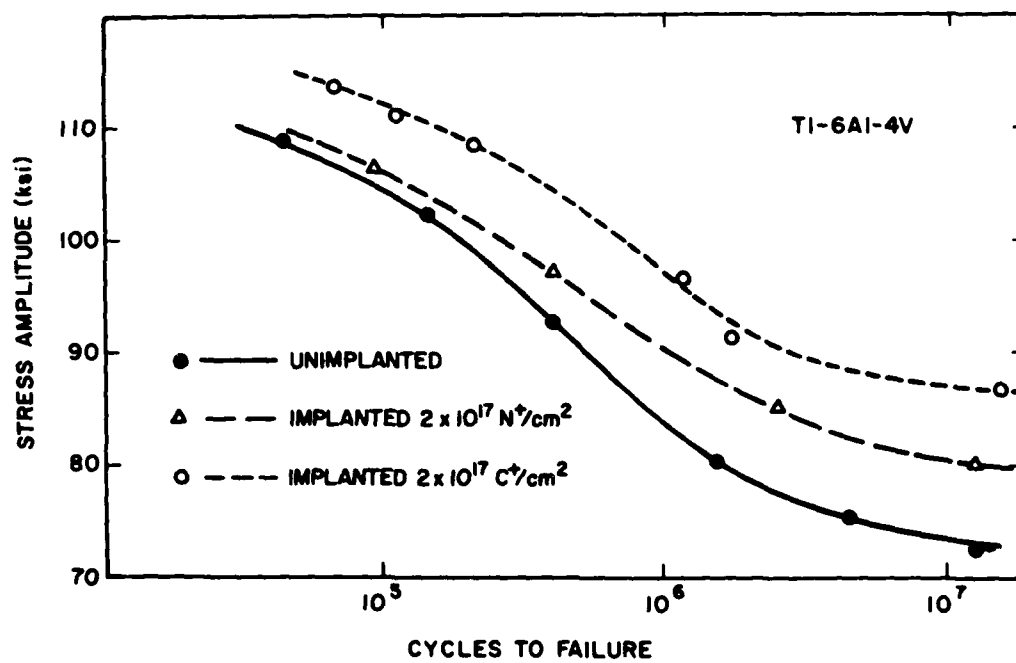Ti-6Al-4V for various conditions as indicated during rotating beam fatigue
testing [51].

Fig. 25   Cycles to failure vs implanted carbon dose for mill
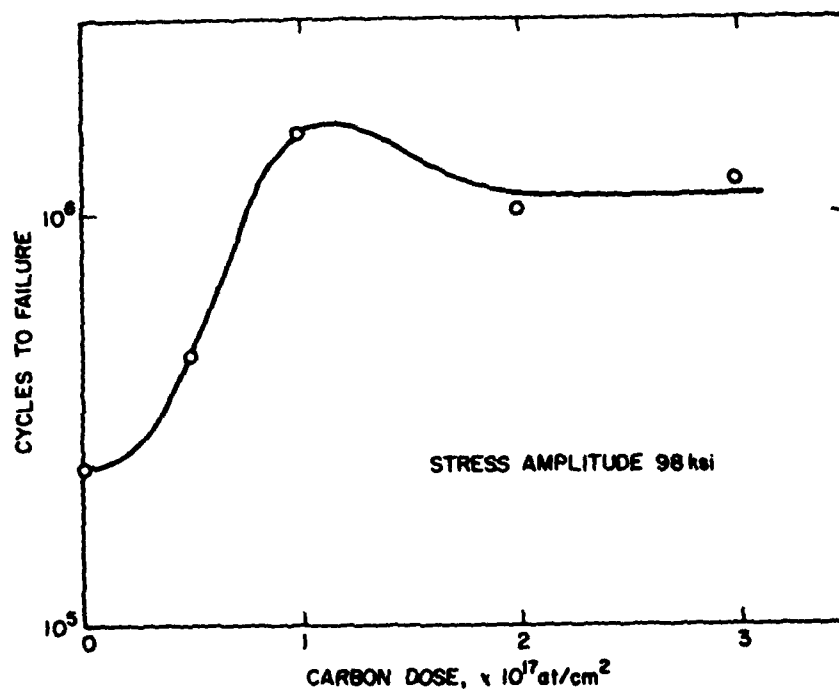annealed Ti-6Al-4V [51].

STRESS AMPLITUDE 98 ksi

CYCLES TO FAILURE

CARBON DOSE, $\times 10^{17}$ at/cm^2

Fig. 26   Effect of ion plating on the tensile strength of stainless
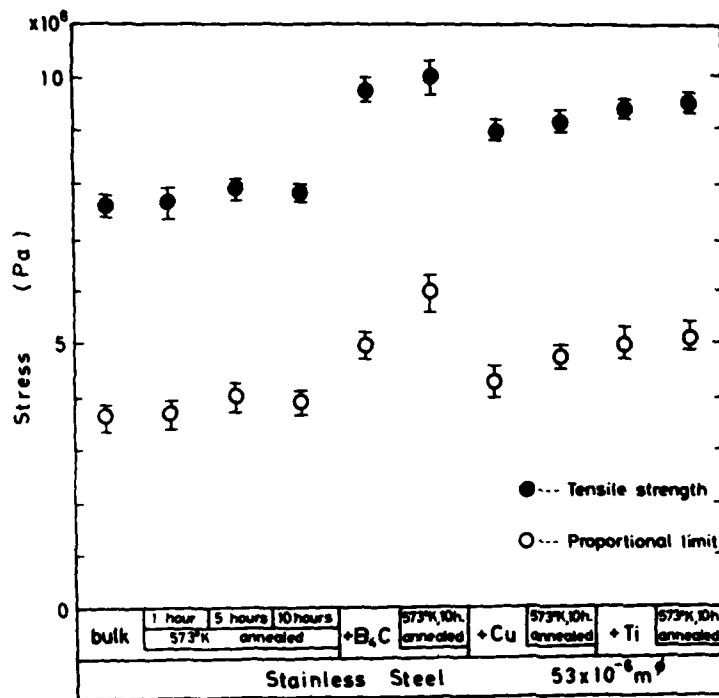steel wire, for various plated coatings as shown [38].

Fig. 27   Effect of ion plating on the tensile strength of tungsten wire, for various plated coatings as shown [38].
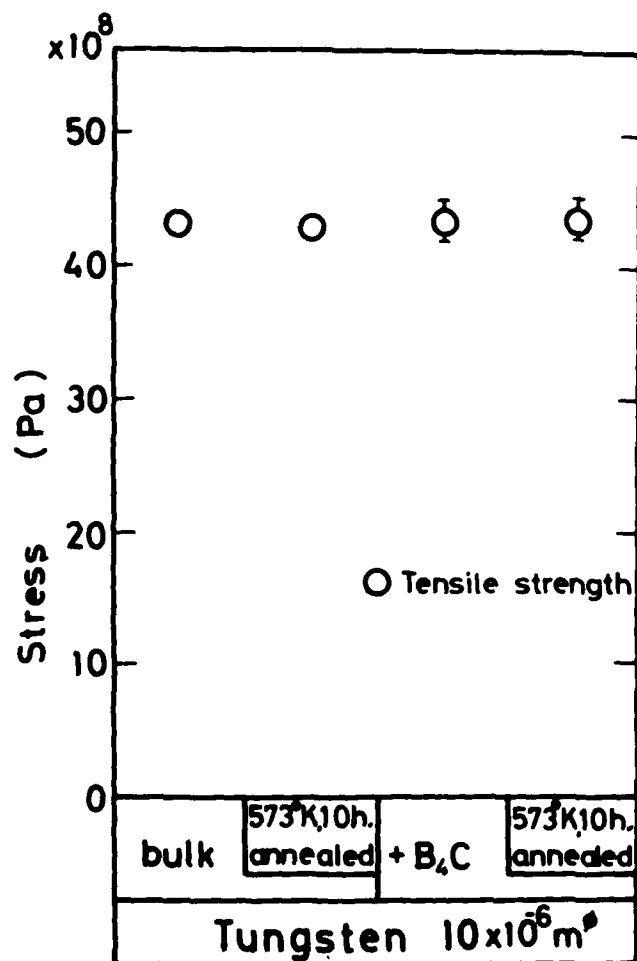
Fig. 28   Average surface loss vs. test time for turbine test blades
of   ECY-768   alloy(66CO-24Cr-10Ni-0.1Al)   and   with   CoCrAlY   coating
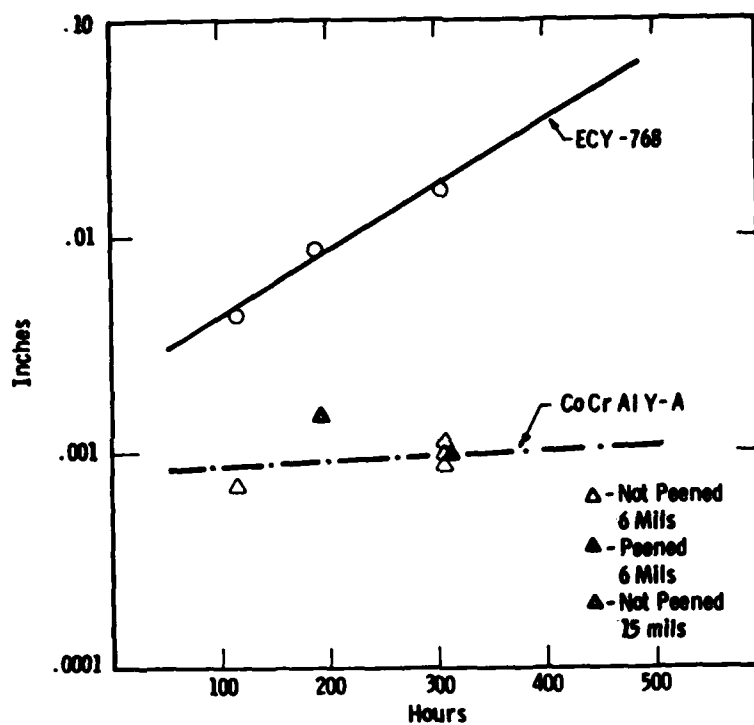(63Co-24Cr-13Al-0.3Y tested at 1173K with 10ppm sodium in the fuel [56].

Fig. 29   Corrosion tests of uranium at 373K in 1 atm of water vapour
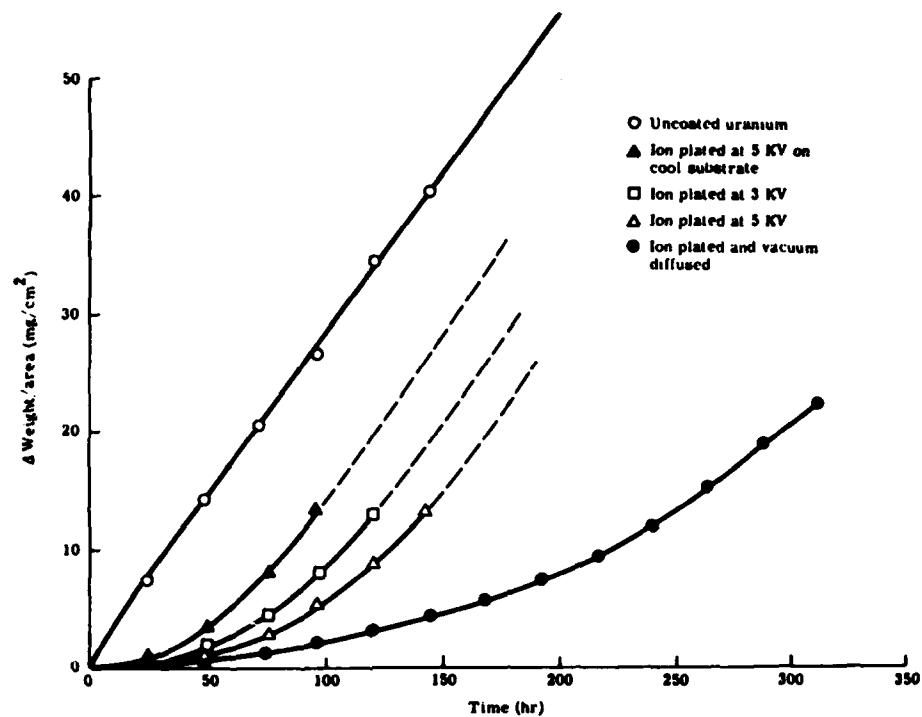pressure for different plating conditions [61].

Fig. 30   Oxide thickness as a function of time for iron (Fe), bulk
alloy Fe-18Cr and surface alloy Fe-24Cr(s), oxidised at 593K in ambient air
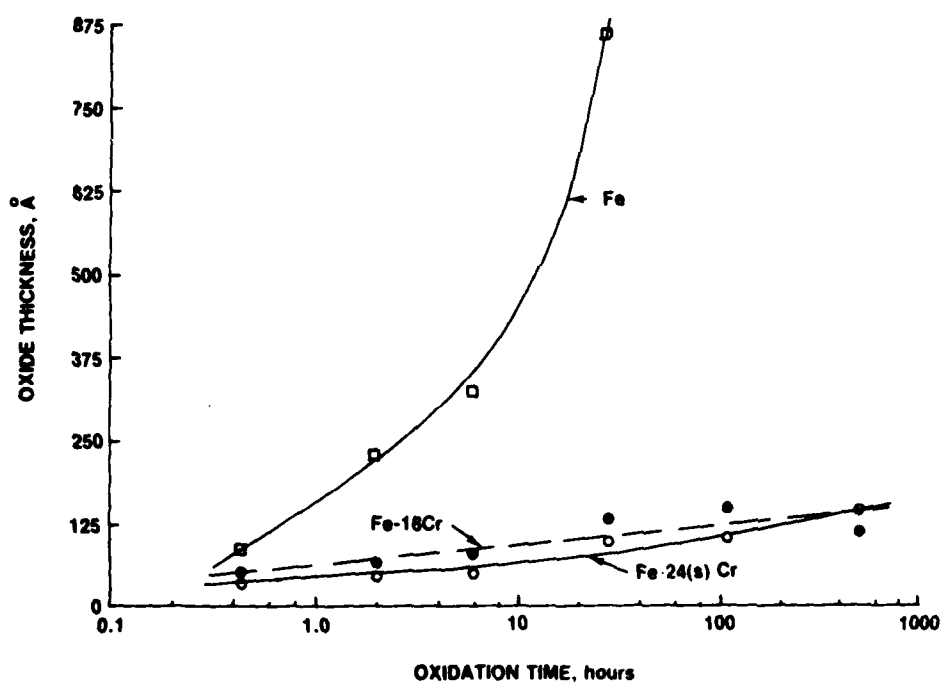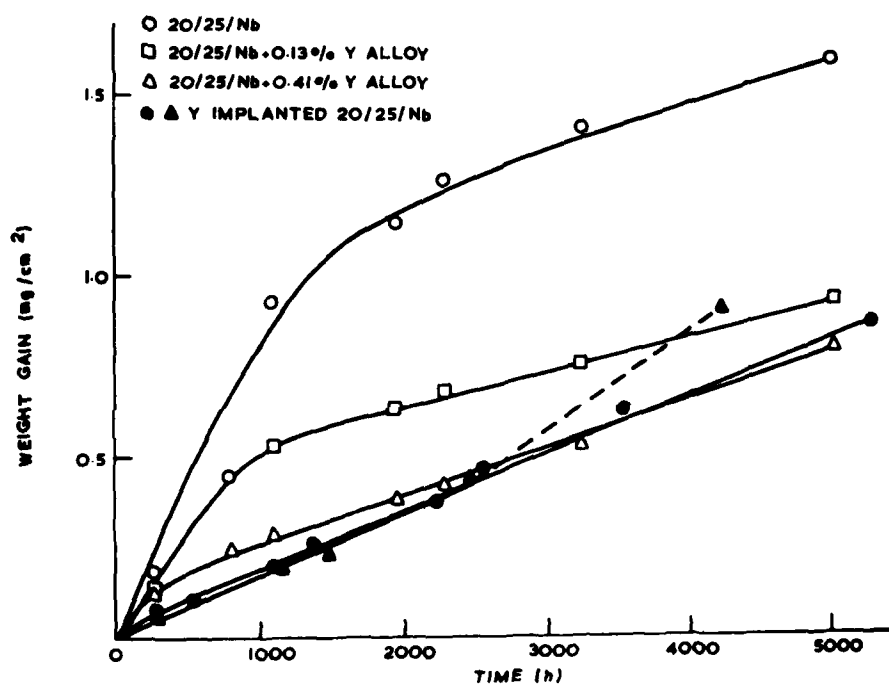environment [26].

Fig. 31   Comparison of the effect of yttrium as an alloy addition or implanted only into the surface upon the oxidation of a 20/25/Nb stainless steel in CO_2 at 1073K [1].

- O 20/25/Nb
- □ 20/25/Nb+0.13% Y ALLOY
- △ 20/25/Nb+0.41% Y ALLOY
- ● ▲ Y IMPLANTED 20/25/Nb

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Ion plating	Corrosion	1103
Ion implantation	Wear	1113
Evaporation	Fatigue (materials)	
Sputtering		
Microstructure		

16. Abstract

    The combination of properties required from modern materials has led to the design of systems in which the bulk physical properties are obtained from the main structure and the required surface properties are provided by a surface coating.

    One important class of techniques for coating surfaces is Physical Vapour Deposition (P.V.D.). This class includes the processes of Evaporation, Sputtering, Ion-Plating and Ion-Implantation, by which a wide variety of materials can be applied to different substrates.

PF 65

16. Abstract (Contd)
A review is given of the above P.V.D. processes and the resultant coating morphologies. Areas of present and future applications are discussed for which surface governed properties such as corrosion, wear, bearing and tool life, fatigue life and surface hardening can be beneficially modified.

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